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LABORATORY EXERCISES
GENERAL CHEMISTRY
—
HOPKINS AND NEVILLE

REVISED EDITION



Class Q D 45

Book H 752

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LABORATORY EXERCISES

AND

PROBLEMS IN GENERAL CHEMISTRY

BY

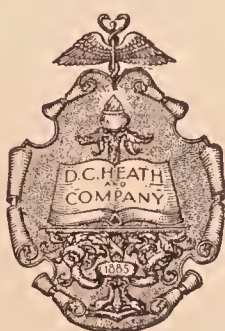
B SMITH HOPKINS

PROFESSOR OF CHEMISTRY, UNIVERSITY OF ILLINOIS

AND

HARVEY A. NEVILLE

ASSOCIATE PROFESSOR OF CHEMISTRY, LEHIGH UNIVERSITY



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LIST OF SUPPLIES

The following is an estimate of the quantities of material and apparatus advisable to purchase for a class of ten students who perform all the experiments in the manual. The amounts listed are generous to allow for waste of chemicals and reasonable breakage of apparatus.

In certain experiments requiring the use of the analytical balance or other limited equipment it will be found advisable not to have all the students perform such experiments in the same period.

GENERAL APPARATUS

- 10 asbestos mats, 10 cm. $\times$ 10 cm.
- 1 balance, analytical, with weights and rider for weighing from 0.1 mg. to 100 g.
- 2 balances, platform, with weights for weighing from 0.1 g. to 1000 g.
- 1 barometer
- 10 brushes, test-tube
- 10 burners, Bunsen
- 10 clamps, iron, small
- 10 clamps, iron, large
- 1 gross corks, assorted sizes
- 1 set cork borers, 6 in set
- 10 crucibles, iron, diam. 4 cm.
- 24 crucibles, porcelain, #00
- 10 cylinders, porcelain, unglazed, diam. 3 cm.
- 24 dishes, porcelain, evaporating, 8 cm.
- 6 electrodes, carbon, diam. 5 mm.
- 10 files, triangular, 15 cm.
- 10 pr. forceps, iron, 10 cm.
- 15 squares gauze, iron wire, asbestos center, 12 cm. $\times$ 12 cm.
- 10 holders, test-tube
- 5 magnets, horse-shoe, small
- 5 magnifiers or lenses

- 10 meter sticks, graduated in inches on reverse side
- 10 mortars, porcelain, with pestle, 9 cm.
- 10 pks. paper, filter, qualitative, 13 cm.
- 2 pks. paper, filter, qualitative, 20 cm.
- 12 vials each, paper, litmus, red and blue
- 12 pinch cocks, Mohr's, medium
- 1 m. platinum wire, #25
- 10 racks, test-tube
- 10 sponges
- 10 spoons, iron, deflagrating, diam. of bowl 1 cm.
- 10 spoons, porcelain, spatula end, 15 cm.
- 10 stands, filter
- 10 stands, iron, with 2 rings
- 1 kg. stoppers, rubber, assorted sizes #0-6, one- and two-hole
- 10 thermometers, chemical, 0°-250° C.
- 10 triangles, pipestem, to support crucible #00
- 10 tripods, iron, diam. of ring 10 cm.
- 10 troughs, gas-collecting — unless sinks are adapted to this purpose
- 10 m. tubing, rubber, for Bunsen burners, inside diam. 6 mm.
- 7 m. tubing, rubber, soft, inside diam. 5 mm.
- 2 voltmeters, student type
- 10 wing tops for Bunsen burners

GLASSWARE

- 24 beakers, 100 cc.
- 24 beakers, 150 cc.
- 24 beakers, 250 cc.
- 20 beakers, 400 cc.
- 12 beakers, 600 cc.
- 12 boats, porcelain, small
- 50 bottles, wide-mouth, 250 cc.
- 12 bottles, wide-mouth, 500 cc.
- 10 burettes, 50 cc.
- 15 flasks, Erlenmeyer, 250 cc.
- 15 flasks, Florence, 100 cc.
- 15 flasks, Florence, 500 cc.
- 12 funnels, 60°, 6.5 cm.
- 12 funnels, 60°, 9 cm.
- 12 graduates, 10 cc. or 25 cc.

12 graduates, 100 cc.
12 pipettes, 10 cc.
50 plates, glass, 8 cm. $\times$ 8 cm.
12 plates, cobalt glass, 8 cm. $\times$ 8 cm.
0.5 kg. rods, glass, diam. 4 mm.
12 tubes, calcium chloride, 15 cm.
12 tubes, test, hard glass, 15 cm. $\times$ 15 mm.
12 tubes, test, soft glass, 18 cm. $\times$ 25 mm.
15 doz. tubes, test, soft glass, 15 cm. $\times$ 20 mm.
15 tubes, thistle, 25 cm. stem, 5 mm. diam.
10 tubes, U, diam. 1 cm.
4 m. tubing, hard glass, combustion, diam. 15 mm.
2 kg. tubing, soft glass, medium walls, outside diam. 6 mm.
24 watch glasses, diam. 8 cm.

This list does not include reagent bottles for desks and side shelf. The size and number of such bottles will be dependent upon the number of students taking the course.

CHEMICALS

0.5 kg. acid, acetic, 30%
100 g. acid, fluosilicic
5 kg. acid hydrochloric, sp.gr. 1.19
5 kg. acid, nitric, sp.gr. 1.42
100 g. acid, perchloric
5 kg. acid, sulfuric, sp.gr. 1.84
50 g. acid, tartaric
2 l. alcohol, ethyl, 95%
25 g. agar, flake
100 g. aluminum, sheet or wire
100 g. aluminum chloride
0.5 kg. aluminum sulfate, cryst
200 g. ammonium carbonate
0.5 kg. ammonium chloride
5 kg. ammonium hydroxide, sp.gr. 0.9
50 g. ammonium molybdate
100 g. ammonium nitrate
100 g. ammonium oxalate
100 g. ammonium sulfate
50 g. antimony, powd.

100 g. antimony trichloride
100 g. arsenic trioxide
200 g. barium chloride
100 g. barium nitrate
25 g. bismuth
50 g. bismuth trichloride
100 g. bromine
50 g. cadmium nitrate
2 kg. calcium carbonate, marble chips
100 g. calcium magnesium carbonate, dolomite
0.5 kg. calcium chloride, anhydrous, granular
100 g. calcium fluoride, powd.
100 g. calcium nitrate
1 kg. calcium oxide, quicklime, in tin can
0.5 kg. calcium phosphate, bone ash
0.5 kg. calcium sulfate, gypsum, powd.
1 kg. calcium sulfate, plaster of Paris, fine
0.5 kg. carbon disulfide
0.5 kg. charcoal, animal
100 g. charcoal, wood, powd
200 g. chromic anhydride
100 g. chromium nitrate
100 g. cobalt nitrate
0.5 kg. copper sheet, 1 mm. thick
1 kg. copper turnings
1 spool copper wire #24
100 g. cupric chloride
100 g. cupric nitrate
100 g. cupric oxide, powd.
1 kg. cupric sulfate, blue vitriol
0.5 kg. ether
200 g. glucose
200 g. hydrogen peroxide, 3%
50 g. iodine, resublimed
200 g. iron filings
200 g. iron nails, small
1 spool iron wire, piano
100 g. iron chloride, ferric
100 g. iron oxide, ferric
100 g. iron oxide, magnetic
200 g. iron sulfate, ferrous

- 1 kg. iron sulfide, ferrous
- 200 g. lead filings
- 200 g. lead nitrate
- 100 g. lead dioxide
- 100 g. lead monoxide, litharge
- 100 g. lead tetroxide, red lead
- 50 g. magnesium powder
- 50 g. magnesium ribbon
- 200 g. magnesium carbonate
- 200 g. magnesium chloride
- 0.5 kg. magnesium sulfate, Epsom salt
- 0.5 kg. manganese dioxide, powd.
- 100 g. manganese nitrate
- 200 g. mercury
- 50 g. mercuric chloride
- 100 g. mercuric nitrate
- 200 g. mercuric oxide
- 100 g. mercurous nitrate
- 10 g. methyl orange
- 100 g. nickel nitrate
- 0.5 kg. paraffin, solid
- 10 g. phenolphthalein
- 50 g. phosphorus, red
- 50 g. phosphorus, white
- 0.5 kg. potassium aluminum sulfate, alum
- 100 g. potassium bromide
- 200 g. potassium carbonate
- 0.5 kg. potassium chlorate
- 200 g. potassium chloride
- 0.5 kg. potassium dichromate
- 100 g. potassium ferricyanide
- 100 g. potassium ferrocyanide
- 0.5 kg. potassium hydroxide, sticks
- 100 kg. potassium iodide
- 0.5 kg. potassium nitrate
- 200 g. potassium permanganate
- 100 g. potassium sulfate
- 200 g. potassium thiocyanate
- 100 g. silica, fine, infusorial earth or kieselguhr
- 100 g. silver nitrate
- 50 g. sodium

100 g. sodium ammonium phosphate, microcosmic salt
0.5 kg. sodium bicarbonate, baking soda
0.5 kg. sodium carbonate, anhydrous
2 kg. sodium chloride, table salt, fine
0.5 kg. sodium chloride, rock salt, coarse
200 g. sodium dichromate
1 kg. sodium hydroxide
0.5 kg. sodium nitrate
200 g. sodium nitrite
100 g. sodium peroxide
200 g. sodium phosphate, secondary
200 g. sodium potassium tartrate, Rochelle salt
0.5 kg. sodium sulfate, Glauber's salt
200 g. sodium sulfite
0.5 kg. sodium tetraborate, borax
200 g. sodium thiosulfate
200 g. starch
100 g. strontium nitrate
0.5 kg. sugar, cane, granulated
0.5 kg. sulfur, flowers
0.5 kg. sulfur, roll
200 g. tin, granulated
100 g. tin chloride, stannic
100 g. tin chloride, stannous
1 kg. zinc, granulated
0.5 kg. zinc, sheet
100 g. zinc nitrate
50 g. zinc oxide
100 g. zinc sulfate
0.5 kg. Wood's metal

A very few articles of common household use, such as lard, calico, wool yarn, and baking powder are not included in the above list.

TO THE TEACHER

This manual provides a sufficient number of experiments for one year of *General Chemistry* laboratory, including an introduction to qualitative analysis. Ordinarily the exercises in Part I will be performed during the first semester, those in Part II during the second semester.

If only one semester of laboratory work is given in *General Chemistry*, it is suggested that certain supplementary exercises may be chosen from Part II.

In courses designed for students who enter college with credit in chemistry, it may be advisable to omit some of the simpler exercises of Part I and select further experiments of general interest from Part II.

LABORATORY EXERCISES

IN GENERAL CHEMISTRY

PART I

THE NON-METALS

GENERAL LABORATORY RULES

The following points are set down for the guidance of the student. Each item is important and must be strictly observed.

1. Apparatus and surroundings must be kept clean.
2. Return bottles of reagents to their proper places on desk and side shelf immediately after using.
3. **Never** return any material to the reagent bottles.
4. Do not use more material than necessary. Chemicals are expensive and results are often obscured by an excess.
5. Do not discard anything you have obtained until you are sure you have no further use for it. Throw solids into waste jars and liquids into the sink. Acids should be washed out of pipes with a large amount of water.
6. Read the label carefully before taking anything from a bottle. Using the wrong material may result in serious injury. **Never** carry side shelf bottles to your desk. Use clean test tubes for liquids and small squares of paper for solids unless otherwise directed.
7. When heating a substance in a test tube be careful not to aim it at anyone who is near.
8. Exercise particular care in handling hot apparatus, strong acids and alkalies, inflammable substances (warning will be given), and glass tubing. A glass tube should be held in a towel when it is being pushed through a stopper. In case of personal injury, however slight, consult an instructor at once.
9. Study the assignment **before** coming to the laboratory and plan your work for best efficiency. Unless this is done the required amount of work can not be done in the laboratory

period. It is often possible to perform two experiments simultaneously. Be prepared to discuss with your instructor what you are going to do and why.

10. Laboratory notes on an experiment must be written **in your notebook** as you perform that experiment. The notebook is not to be taken from the laboratory except by special permission.
11. Your work must be performed independently, but do not hesitate to ask your instructor for advice. Use your textbook for reference. Above all, **be awake — think!**
12. Just before leaving the laboratory make sure that the gas and water are turned off, that all bottles are in place, that your desk top is clean.

SUGGESTIONS FOR WRITING LABORATORY NOTES

1. Carefully note the title and subtitle of the exercise, realizing that each experiment is intended to illustrate some important chemical fact or principle. Forget for the moment that this experiment has previously been performed by others and feel that for you it is an original scientific research — a “question put to nature.” With this point of view you will see the importance of making accurate observations and careful conclusions.
2. Record what you do without repeating exact directions; a simple drawing may save a description of the apparatus. Observe and record **immediately** any changes that occur. Write out your conclusions from the phenomena observed. When any chemical change is involved **write the equation** for the reaction. Answer in your write-up all questions either direct or implied by (?). In all be **brief, concise** and **to the point** of the experiment.
3. Since these are **notes** and not literary essays, emphasis is placed upon the accuracy and detail of your record rather than upon its form. Use the left-hand side of the page for drawings and calculations if you so desire. If part or all of an experiment is unsatisfactory, mark “Repeated” across the old record so it will not be graded and rewrite that portion. Neatness here is of secondary importance to clarity; yet a reasonable degree of neatness is required in your notes and experimentation.

4. Notes or data, such as weighings, recorded on scraps of paper will be taken up by the instructor and destroyed. Make all records in your notebook directly and immediately. It is both unscientific and inefficient to delay or transfer records of an observation. Whenever possible arrange your data in the form of a table so you may easily draw comparisons and for the convenience of your instructor.
5. Use your textbook for reference constantly in performing and writing up laboratory experiments. Textbook references are given in this manual to *General Chemistry* by B. Smith Hopkins, D. C. Heath and Company, 1930. Read and follow the "Suggestions on How to Study Chemistry," Textbook: vii and viii.
6. A FORMAL REPORT will be required on certain quantitative experiments. For such experiment a record of data is made in the notebook as usual and a copy of this is taken home where a more elaborate write-up is prepared. This is to be enclosed in a manila paper cover and handed in before a date announced by the instructor. In this formal report should be included a complete statement of the purpose and method of procedure in the experiment, a drawing of the apparatus, results (data in tables), calculations and conclusions. Neatness and arrangement as well as accuracy are important features of this report.

PROCEDURE FOR THE FIRST LABORATORY PERIOD

1. Check the apparatus in the desk assigned by your instructor. You should learn at this time the names of any unfamiliar pieces of apparatus. If anything is missing note it on the card, have this countersigned by your instructor, and obtain the articles at the storeroom. When your outfit is complete sign the list and return it to the storeroom.
2. Read carefully the **General Laboratory Rules**. Read them again at home and as often as necessary.
3. Proceed with Exercise 1. Practice working glass tubing according to the instructions and when sufficient technique has been acquired prepare a wash bottle as in Figure 4. Submit this to your instructor and, if approved, fill it with distilled water and keep it in your desk for use when directed. No record of this exercise is required.

EXERCISE 1

THE USE OF GLASS TUBING

(a) **To Cut Glass Tubing.** — By means of a triangular file make a transverse scratch upon the glass tubing at the desired point. Hold the tube so that the ends of the thumbs are together on the tube opposite the scratch as in Figure 1. Press outward with the thumbs and pull gently in the opposite direction with both hands. To cut wider glass tubing a deep scratch should be made completely around the tubing and a crack started by touching the scratch with the red-hot end of a glass rod.

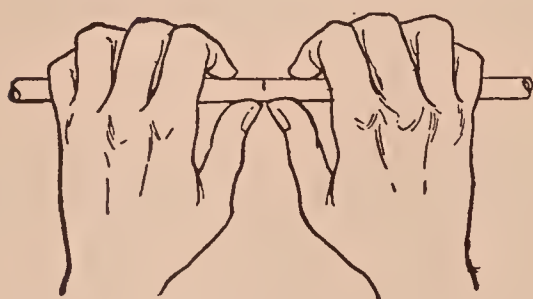


FIG. 1

touching the scratch with the red-hot end of a glass rod.

(b) **To Fire-Polish Glass.** — Always round off the edges of freshly cut glass tubing or glass rod in order to avoid injury to your hands and to prevent cutting the rubber stopper or rubber tubing into which the glass may be inserted. Fire-polishing is accomplished by rotating the end of the glass tube or rod in a Bunsen burner flame until the sharp edges become smooth. Why does this occur? If this treatment is continued too long with small glass tubing the end of the tube will close completely.

(c) **To Bend Glass Tube.** — Attach a “wing-top” to a Bunsen burner, close the air holes at the bottom, and light the burner. Hold a piece of tubing lengthwise in the yellow luminous flame, as in Figure 2, not across the flame, rotating the tubing slowly to insure uniform heating. Continue heating the tube in this manner until it is soft enough to bend of its own weight, then remove it from the flame and at once bend it to the desired angle. Try to produce a bend that will coincide

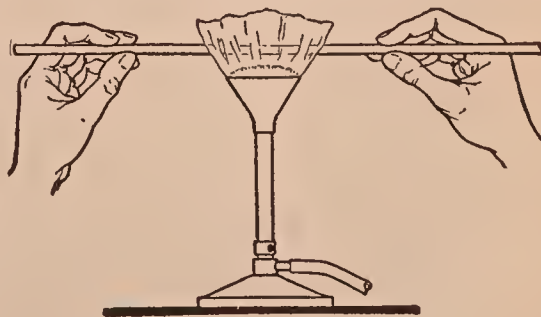


FIG. 2

with some angle drawn on a piece of paper. The hot glass should not come in contact with the varnished surface of the desk but should be suspended in the air or laid upon a piece of asbestos to cool.

The bends *A* and *B* in Figure 3 are correctly made; *C* and *D* are unsatisfactory because of the constriction at the bend and because they are more likely to break.

Bends such as *C* and *D* result from non-uniform heating of the glass, heating the tube through too short a length, bending the tube while it is in the flame, or forcing the glass to bend before it has been heated to the required softness.

(*d*) **To Make a Jet.** — Remove the wing-top, light the burner, and open the air holes at the bottom to produce a hot, non-luminous flame. Heat a piece of glass tubing at a distance of 2 or 3 inches from one end, rotating the tubing in the hot flame until the glass begins to collapse. Remove the tubing from the flame and pull the ends apart in a straight line until the capillary has the desired size. When the glass is cool, carefully cut the capillary and fire-polish both ends of the jet.

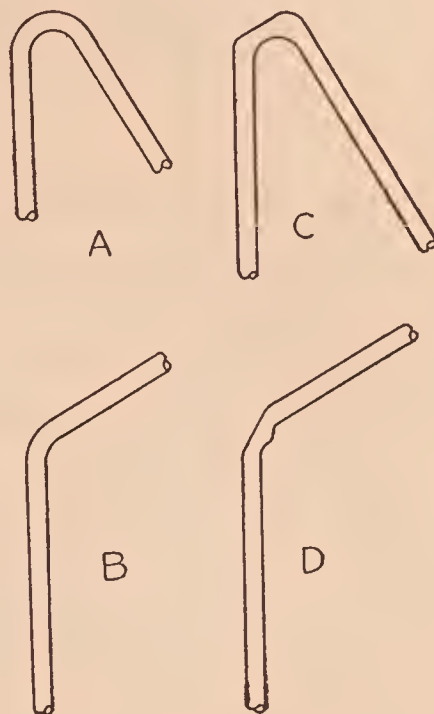


FIG. 3

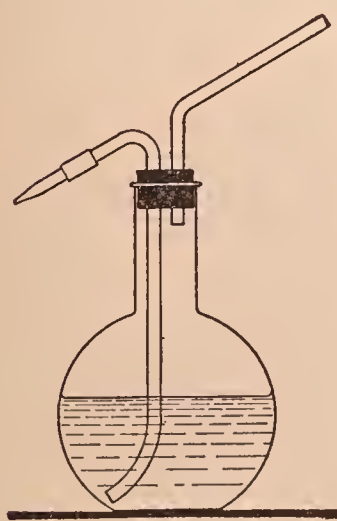


FIG. 4

(*e*) **To Insert a Glass Tube in a Stopper.** — Beginners in the chemical laboratory probably suffer more injuries from carelessness in this simple operation than from any other single cause. There is no danger from this source if the following precautions are observed: See that both ends of the tube are properly fire-polished. Be sure that the tube is cold, and wet both tube and stopper. Push the tube through the hole in the stopper with a screw-like motion. If the tube is fragile or has a bend in it, protect your hand with a towel. Do not exert a strain upon a bend in the tube.

(*f*) **Construction of a Wash Bottle.** — Obtain a two-hole rubber stopper from the storeroom to fit the 500-cc. Florence

flask, prepare the other parts, and assemble a wash bottle as in Figure 4.

Before coming to the next laboratory class read carefully **Suggestions for Writing Laboratory Notes** and study Exercise 2.

EXERCISE 2

MEASUREMENT — THE METRIC SYSTEM

Textbook: 54-55

The metric system of measurement is used for most scientific work and is gradually coming into general use. It has three fundamental units — the **meter**, the **liter**, the **gram**. These units are interrelated and all multiples and submultiples are decimal. **Refer to table near back of this book.**

(a) **Length.** — Examine a meter stick on both sides. Calculate the percentage (of a yard) by which a meter exceeds a yard. Measure a length of more than two feet in both inches and centimeters and from these values calculate the number of centimeters in an inch.

(b) **Volume.** — A liter is the volume of a cube having edges $1/10$ (one-tenth) meter in length. Draw a liter cube and label it to show the relation of the length of edge to volume. How many cubic centimeters (cc.) are there in a liter?

Measure the volume of an ordinary test tube by means of the graduated cylinder. Mark with gummed labels the test tube levels that correspond to 10 cc. and 20 cc. This tube may be used in your work for approximate measurements.

(c) **Weight and Density.** — Weigh an empty beaker on the laboratory scales. Fill it about half-full of water and weigh again. Measure the water and calculate the weight of 1 cc. of water and of 1 liter of water. Label the cube drawn above with the weight of water required to fill it. Your drawing now demonstrates that, in the metric system, the units of length, volume and weights are related.

If the density of concentrated sulfuric acid is 1.8 (density of water is 1), what is the weight of the contents of the bottle of that acid on your desk? Its volume is one-fourth liter.

Determine the density of some solid as follows: Carefully weigh about 20 g. of some insoluble material — *e.g.*, broken glass tubing, marble chips, ferrous sulfide, or iron nails. Fill

the 100-cc. graduate with water to a definite mark (read opposite the bottom of the curved water surface or meniscus), add the weighed material and note the increase in volume. From this data calculate the density (specific gravity) of the material to the second decimal place. State in words and by a mathematical formula the relationship of density, weight and volume.

Complete the following by supplying the correct number:

0.8 meter	=	centimeters
2364 millimeters	=	meters
1.75 grams	=	milligrams
1250 cc.	=	liters
2.4 kg. of water	=	cubic centimeters

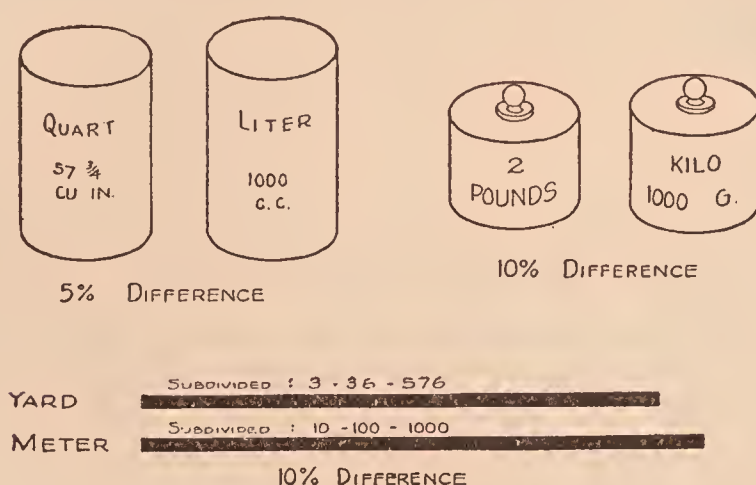


FIG. 5

LABORATORY TECHNIQUE

It is very important to form correct habits in the laboratory. The best method for performing any operation should be learned by the student when that operation is first encountered, and this method should be used thereafter on all occasions. A few general instructions were given under **General Laboratory Rules**. Refer to these from time to time until you are sure you are observing all of them as a matter of habit. Further definite instructions for correct procedures will be presented in the exercises as they are needed; the following processes of manipulation should be learned at this point for immediate use.

(a) **Removing Solids from Bottles.** — The required amount of a solid should be removed from the bottle by means of the hollow glass stopper as indicated in Figure 6. If the solid is to be weighed on the laboratory scales, two pieces of paper of equal

weight are placed, one on each platform of the scales, the weights are placed on the right-hand platform or the rider is moved to the right, and the solid is then slowly poured upon the paper on the left-hand platform by tilting and gently rotating the stopper until the scales are balanced.

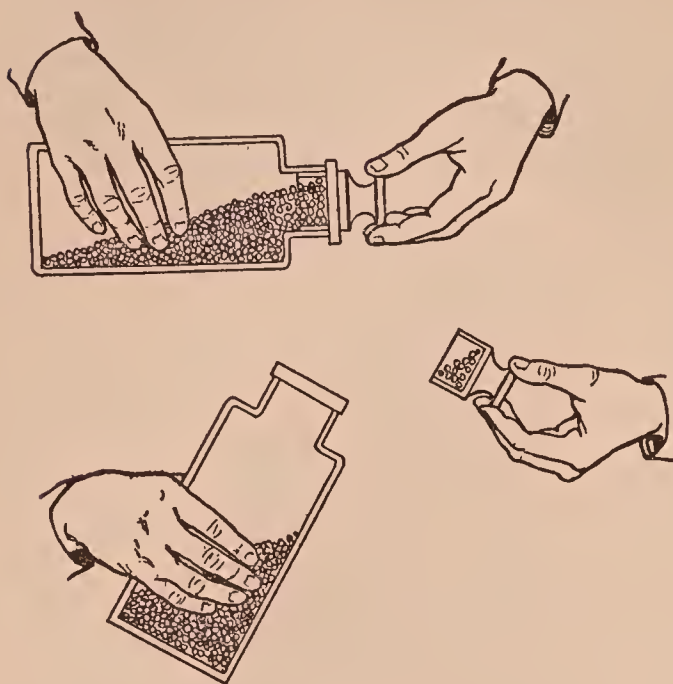


FIG. 6

(b) **Pouring Liquids from Bottles.** — When using a reagent from a glass-stoppered bottle, remove the stopper by taking it between the fingers at the back of the right hand as shown in Figure 7a. The presence of the stopper does not interfere with the use of that hand for

holding the bottle. **Never** lay the stopper down as it will become contaminated and may, when returned to the bottle, make the contents unfit for use. In pouring from a bottle the neck of the bottle should **touch** the edge of the receiving vessel (Fig. 7b). This prevents the liquid from running back

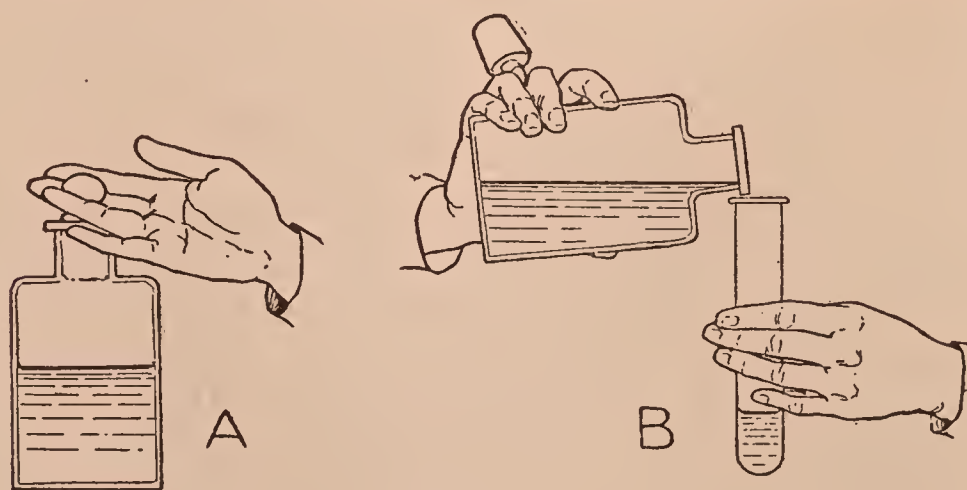


FIG. 7

on the side of the bottle and is especially important in the handling of strong acids or other corrosive liquids.

Never dip a stirring rod, pipette, platinum wire or other apparatus into a reagent bottle. To obtain a drop of the reagent on a stirring rod, pour some of the reagent into a watch glass or test tube and dip the rod into this.

(c) **Filtering.** — Prepare the filter paper by folding it along its diameter and then folding it again at a right angle to the first fold (Fig. 8). Open the paper to form a cone, fit this in a funnel, and wet the paper with a few cubic centimeters of distilled water to cause it to stay in place. Pour out any excess water before using the filter.

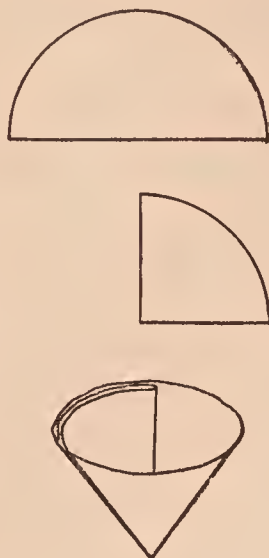


FIG. 8

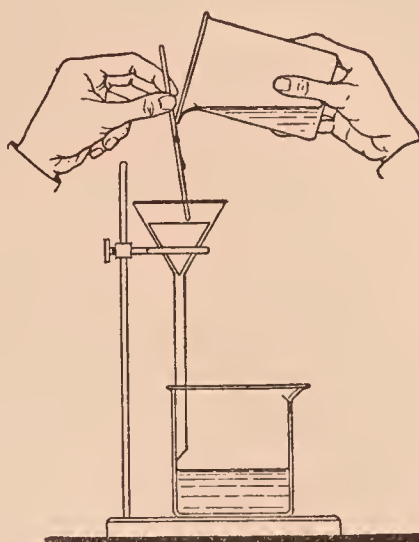


FIG. 9

Fig. 9 shows the correct arrangement for filtering. The liquid should be poured slowly along the stirring rod and the filter paper should not become too full. The

filtrate, passing through the stem of the funnel, should run down the side of the receiving vessel and should not be allowed to fall in drops because this would cause spattering.

If the **precipitate** is very finely divided it may run through the filter paper at first. If the first portion of the cloudy filtrate is again filtered it will probably come through clear since the filter paper becomes more effective after some of the precipitate has been collected in the paper.

If a precipitate is heavy and settles well, most of the liquid may be separated from it by carefully pouring the liquid away from the solid. This process is called **decantation**. For a more complete separation the liquid may be decanted through a filter.

(d) **Heating Materials in Glass and Porcelain.** — Thick-walled vessels of glass or porcelain must **never** be heated. When heating apparatus of glass or porcelain remember to raise the temperature gradually. Sudden heating or cooling may cause the apparatus to crack.

Beakers, flasks and evaporating dishes should be heated on a wire gauze and not in contact with the direct flame.

When heating a liquid in a test tube, raise the temperature gradually and uniformly throughout the depth of the liquid to avoid the sudden formation of vapor below the surface of the liquid. Uniform heating may be accomplished by passing the

tube back and forth through the flame and agitating the contents of the tube, or by waving the flame along that part of the tube occupied by the liquid.

EXERCISE 3

PROPERTIES OF MATTER — CHEMICAL AND PHYSICAL CHANGE

Textbook: 18-22

(a) Examine a clean piece of copper wire or copper foil and note its properties. Hold the piece of copper by means of a pair of forceps in the outer flame of the Bunsen burner until the copper is red-hot. Remove the copper from the flame and observe it while it cools in the air (?). Compare the properties of the product upon the surface of the copper with the properties of the original copper.

Repeat the experiment with a platinum wire and with a piece of magnesium ribbon. Tabulate the properties of each metal before and after heating. State in each case whether a chemical or physical change was brought about by the application of heat to the metal.

Place the piece of platinum wire, a clean piece of copper and a piece of magnesium ribbon in separate test tubes and add 3 or 4 cc. of dilute hydrochloric acid to each. Is there evidence of chemical change in any of these? Repeat this part of the experiment using a few drops of concentrated nitric acid with each metal. Describe the results and state the evidence for any chemical changes observed. Wash the acid from the piece of platinum wire and preserve the wire for subsequent use. Summarize the results you have obtained to show the differences in the chemical properties of the three metals.

Define briefly and illustrate: *physical property, chemical property, physical change, chemical change.*

(b) Obtain about 2 g. of sodium carbonate, note its properties (color, taste, etc.) and dissolve it in 30 cc. of water, warming the water to hasten solution. Divide this solution into three equal portions and proceed with the separate portions as follows:

1. Evaporate this portion to dryness in a porcelain dish and compare the residue with the original substance.

2. Add dilute hydrochloric acid, a few drops at a time with

stirring, to this portion until no further action is observed and evaporate this portion to dryness in a porcelain dish. Examine this residue and compare it with the original solid with respect to appearance and taste.

3. To the third portion add a solution of calcium chloride until no further change is observed. Filter out the precipitate, transfer it to a small beaker and dissolve it in the minimum quantity of dilute hydrochloric acid (?). Evaporate this solution to dryness and examine the residue. It is calcium chloride. State, in the order of their occurrence, the changes which have been brought about in this portion of the sodium carbonate solution. If the *filtrate* obtained in this experiment had been evaporated to dryness, what residue would have been obtained?

Record and learn the formulas for all of the substances used in this exercise. Write the equations for each of the chemical changes observed in this exercise.

EXERCISE 4

ELEMENTS, COMPOUNDS — SEPARATION FROM MIXTURES

Textbook: 24-28

The substances to be found in the laboratory, if pure, may be classified as elements and compounds. Define each class.

Write in your laboratory notebook a brief description of four **elements** found on the side shelf. Two should be **metals** and two **non-metals**. Give name and symbol and such properties as color, form, hardness, etc.

Find two **soluble compounds** and two **insoluble compounds**. Give names and **formulas** and describe briefly. To test solubility take a very small quantity in one-half test tube of water and shake — heat if necessary. A solution must be clear and transparent if not too deeply colored. State the distinction between symbol and formula.

Now place about a gram of a soluble substance and about the same quantity of an insoluble substance (both finely divided) in a small beaker, add 20 cc. of water, heat to boiling and decant through a filter. What is the **residue**? Evaporate the **filtrate** to a small volume and allow to cool. Identify the crystals. Has a chemical change occurred?

Two soluble substances may be separated by **fractional crystallization**: in general, the less soluble one will crystallize first. Referring to the solubility curves in your textbook, is sodium chloride or potassium nitrate less soluble in cold water? In boiling water? Which curve is most influenced by temperature change?

Take 3 g. common salt (sodium chloride, formula?) and 10 g. saltpeter (potassium nitrate, formula?) in a test tube, add 15 cc. of water; warm until the salts are dissolved. Cool the solution by placing the tube in a beaker of cold water. Note the shape of the crystals; taste them. What are they? Pour off the mother liquid into a small beaker and evaporate it slowly until the crystals appear in the hot liquid. Note their form and taste. What are they? As the remaining solution now cools what crystals appear?

Briefly indicate how you could separate two mutually soluble liquids such as alcohol and water.

EXERCISE 5

STUDY OF A MIXTURE

(a) Gunpowder is a mixture of saltpeter (potassium nitrate), sulfur and charcoal (carbon). Grind **separately** to a fine powder 3 g. saltpeter, 0.5 g. charcoal, and 0.5 g. sulfur. Mix these thoroughly on a piece of paper. Place half the mixture in a dry test tube, add 5 cc. of carbon disulfide, shake well (closing test tube with thumb) and filter,* bringing all the solid residue on the filter paper. (*Caution*: Carbon disulfide is very inflammable; do not bring it near a flame.) Catch the filtrate in a watch glass and allow it to evaporate spontaneously.

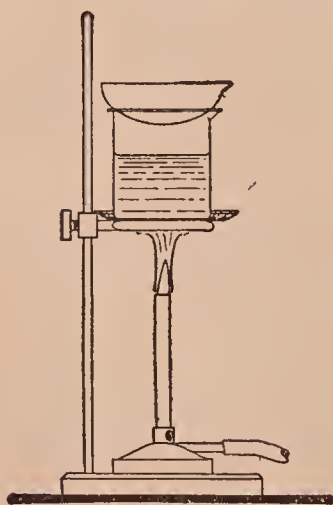


FIG. 10

Allow the residue in the filter paper to dry, then pour 10 cc. of warm water through it, a little at a time. Catch this filtrate in a porcelain dish and evaporate it almost to dryness ~~on a water bath~~ (Fig. 10). Identify the crystals. What is left on the watch glass? On

* Do not wet the filter paper with water in this case.

the filter paper? Carbon is not soluble in any laboratory reagent.

(b) Make a conical pile of the remainder of the gunpowder on an asbestos mat and ignite it by means of a paper fuse or the Bunsen burner. Compare the volume of the residue with that of the gunpowder used. To what form was most of the material converted? List the properties of gunpowder which you think make it useful as an explosive or propellant.

EXERCISE 6

CHEMICAL CHANGE

(a) **Combination of Two Elements.** — Grind together 7 g. fine iron filings and 4^{3 g. 0.5 g.} g. sulfur. Examine some of the mixture with a lens. Is it homogeneous? Spread some of the mixture on a piece of paper, draw a magnet under it or through the mixture and blow away the sulfur (?). State briefly another method by which you could separate the iron and sulfur. (Refer to previous exercise.)

Save out a small portion of the mixture for later comparison, put the remainder in a dry test tube, and while holding the tube in an inclined position apply a vigorous flame to the lower end until the mixture begins to glow. Remove the tube from the flame and notice the progression of the glow through the mass. What is the source of this heat? **One characteristic of a chemical change is the transformation of chemical energy into heat, or vice versa.**

When the tube is cool, break it and examine the product, comparing it with the original mixture. Is it homogeneous? ^{yes} Magnetic? ^{no} Can the two elements now be separated by means ^{no} of CS_2 ? (What has taken place?) In test tubes add a little dilute hydrochloric acid to some of this product and to some of the original mixture. Determine by odor whether the same ^{no} gas is evolved in both cases. **A second characteristic of chemical change is the formation of one or more new substances.**

(If all the mixture had been heated, what weight of iron sulfide would have been formed? **The third characteristic of chemical change is constancy of total weight.**

What is the formula of a compound of the above proportions?

small (Refer to relative (atomic) weights of iron and sulfur. Calculate also the formula of a compound consisting of seven parts iron to eight parts sulfur. See Model Example II.

(b) **Decomposition of a Compound.** — Prepare a tube about 10 cm. long by taking a longer piece of glass tubing, heating it 10 cm. from one end, and drawing the two pieces apart until a closed tube is made. Put a small quantity of mercuric oxide (formula?) into this tube and heat it. Insert a glowing splinter of wood into the tube (?). This is a test for oxygen. What other substance is formed? Note down a few of its properties. Restate the results of this experiment in terms of the three characteristics of chemical change. *legible*

black What change in color is noticed as the mercuric oxide is heated and as it cools? Is this a physical or chemical change? *red physical*

EXERCISE 7.

SOURCES OF OXYGEN

Textbook: 30-33

(a) Air is a mixture of gases, about one-fifth being oxygen. How may oxygen be separated from this mixture? How is oxygen obtained from water?

(b) To prepare oxygen in the laboratory some oxygen compound is usually decomposed by heat as in Exercise 6 (b). Write the formulas for the following substances and obtain samples (about 0.5 g.) from the shelf: ferric oxide; potassium chlorate; sodium carbonate; manganese dioxide; potassium nitrate. Heat each of these separately in tubes prepared as in Exercise 6 (b), or in the small hard glass test tube, and test for oxygen (?). Do you know any other compounds that yield oxygen on heating? What would you call that property which determines whether or not an oxygen compound will liberate oxygen when heated?

(c) Place one-half gram sodium peroxide (storeroom) in a test tube, add a few drops of water and test for the evolution of oxygen (?). Add more water and test the solution with red litmus paper (?). Write a word equation to show the reactants and products in this change. Repeat the equation using formulas.

EXERCISE 8

CATALYSIS

Textbook: 33-34

(a) Place a gram of potassium chlorate in a dry test tube, clamp this on the iron stand in an inclined position and heat carefully with a Bunsen burner until the salt melts. Have ready at hand a small amount of manganese dioxide. Heat the potassium chlorate slowly, just above its melting point, so that bubbles of gas rise very slowly through the liquid. Test for oxygen (?). Remove the flame, add the manganese dioxide and note the effect. Test again with a glowing splinter (?).

(b) Heat the mixture until the potassium chlorate is entirely decomposed. Test (?). Allow it to cool, add 10 cc. of water, shake to disintegrate the residue and pour through a filter paper. Catch the filtrate in a porcelain dish and evaporate it to dryness. Compare it by taste and appearance with potassium chlorate. Does it contain oxygen? How can you prove it?

What is the residue on the filter? How could you prove its identity? Explain its use in this experiment. Can manganese dioxide be made to give up a part of its oxygen (Exercise 7)? Is ferric oxide decomposed by heating (Exercise 7)?

(c) Repeat (a) using ferric oxide in place of manganese dioxide.

EXERCISE 9

PREPARATION OF OXYGEN; COMBUSTION —
FORMATION OF ACIDS AND BASES*Textbook: 33-36*

Make a mixture of 10 g. of potassium chlorate and 3 g. manganese dioxide. Place this in a test tube provided with a one-hole stopper and a delivery tube. Arrange the apparatus as shown in Figure 11. The lower end of the delivery tube must be curved upward so a bottle may be inverted over it. The test tube should not be more than half full of the mixture. Fill four bottles with water, cover with glass plates and invert them in the sink or gas-collecting trough which is partly filled with water.

Place a bottle filled with water over the end of the delivery tube and gently heat the mixture in the test tube. Do not allow the molten contents of the test tube to come in contact with the stopper. Collect four bottles of oxygen in this manner. As each bottle is filled, slip a glass plate over its mouth and set the bottle upright on the desk for use in the following experiments:

(a) **Carbon.** — Wrap a piece of iron wire around a bit of charcoal or place the charcoal in a clean iron deflagrating spoon. Heat this in the Bunsen burner until the charcoal glows and then lower it into a bottle of oxygen (?). Do not drop the charcoal into the bottle. When the charcoal no longer burns, remove it and add 10 cc. of water, shake well and test with blue litmus (?). Pour part of the solution into a test tube and add about 5 cc. of limewater (calcium hydroxide) (?). The precipitate is calcium carbonate, having the same composition as chalk, marble and limestone.

Put 5 cc. of limewater in a test tube and blow your breath through it by means of a glass tube (?). For what gas is limewater a test?

Write equations for the combustion of charcoal; for the addition of water to the product; for the addition of limewater.

(b) **Sulfur.** — Burn some sulfur in the iron spoon in another

bottle of oxygen (?). Notice the odor. Add water, shake and test with blue litmus (?). Test the dilute acids on your desk with litmus. What element is common to all acids? Name two acids found in foods or beverages. *gas*
acid

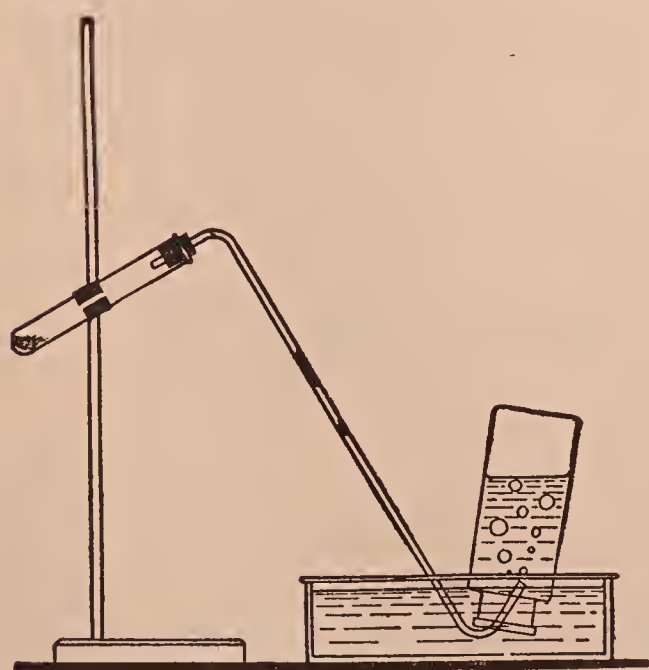


FIG. 11

(c) **Phosphorus.** — (Caution: White phosphorus is kept under water because it burns spontaneously in the air. It must be handled with forceps. If it takes

fire in contact with the flesh it produces very painful burns.)

In a dish of water obtain a small piece of phosphorus from the storeroom. Lay it for a moment on a piece of filter paper

to dry it, then place it in the deflagrating spoon. Ignite the phosphorus by touching it with a slightly warm file or piece of glass tubing and at once lower it into a bottle of oxygen. What is the appearance of the product? Add water and test with litmus (?).

(d) **Magnesium.** — Obtain a piece of magnesium ribbon about 5 cm. long. Hold the ribbon in forceps, ignite it in the Bunsen flame and thrust it into a bottle of oxygen. The light of burning magnesium is very rich in ultra-violet rays. What use is made of magnesium in photography? Examine the product (?). Add water and test with red and blue litmus papers (?). Classify as metals and non-metals the four elements which you burned in oxygen. State the reactions by which:

1. An acid may be formed from a non-metal.
2. A base may be formed from a metal.

In order that these two statements may be true the oxide must, of course, be soluble in water.

Be sure you have written equations for all the chemical changes which you observed in this exercise.

EXERCISE 10

ACCURATE WEIGHING

The instructor will explain the operation of the chemical balance and give directions for its use. Note carefully the following points:

1. The balance is a delicate and expensive instrument; handle it with care. If it does not seem to work properly call your instructor; do not attempt to adjust it yourself.
2. The object to be weighed is placed on the pan at the left of the observer. Begin with larger weights and work down to the correct weighing.
3. Handle weights with forceps only. Do not borrow weights or rider from any other balance.
4. **Never** put anything on the pans or remove anything from them while the beam is unsupported. Do not release the beam very far unless the pans are very nearly balanced.

5. **Chemicals** are not to be placed directly on the balance pan: use glass or porcelain containers.
6. The balance case should be closed while determining the **zero point** or taking the **final** reading of a weighing.
7. In an experiment where several weighings are to be made, the same balance should be used for all.
8. Count weights carefully and check by noting vacant places where weights are kept. The weighings must be recorded **directly** in your notebook — never carry scraps of paper into the balance room.
9. After a weighing is completed, always return weights to their **proper** places and raise the rider on its support. See that the balance case is left in order and its door closed.
10. Observe strictly the directions posted in the balance room.

The instructor will give you an accurately weighed piece of metal for practice weighing. Determine the weight of this to the fourth decimal place (tenth of a milligram); record it with the number of the metal slug and the zero point of the balance in your notebook and submit this record to your instructor.

The Zero Point. — Before making any weighing the “zero point” of the balance must be determined. This is the true center of the swings which the pointer makes on the lower scale. If the balance were in perfect adjustment this zero point would coincide with the center of the scale. This will rarely be the case as the adjustment is affected by many influences. The zero point is found by recording an uneven number of consecutive swings to the left and right. The first and last swings are thus on the same side and the shortening of the swings due to friction is, therefore, neutralized. The average swing for each side is calculated, the difference between these, divided by 2, is the zero point. An example should make this clear:

	<i>Left</i>	<i>Right</i>
Swing — (1)	5.4	(2) 4.2
(3)	5.0	(4) 3.8
(5)	4.6	
Average —	5.0	4.0

Zero point = 5 (left) minus 4 (right) $\div$ 2 = 0.5 to the left. The true center of the swings is hence one-half division on the ivory scale to the left of the center line.

Weighing. — In making the weighing the weights are adjusted until the pointer is swinging equally on both sides of the zero point just found. This may be accurately determined by recording the swings and making a calculation as for the zero point. The zero point of the loaded balance should closely coincide with the zero point when the pans are empty.

The most common error of the beginner is the incorrect expression or addition of the weights. A weighing should be expressed decimally in terms of **grams**. Thus, the following weights: 5 g., 200 mg., 50 mg., 10 mg., rider on 6.4 mg., are recorded as 5.2664 g.

EXERCISE 11

WEIGHT OF A LITER OF OXYGEN — LAW OF CONSTANT COMPOSITION

Quantitative — Formal Report Required

In a clean, dry, hard-glass test tube put a small amount (about 0.3 g.) of the manganese dioxide provided in the balance room. Weigh accurately, expressing the weight to four decimal places. Now add about a gram of potassium chlorate (balance room) and weigh the tube again. The difference between these two weighings represents the accurate weight of what? Why is it not necessary to know the exact weight of the manganese dioxide? Mix the materials by shaking, being careful not to lose any.

Arrange the apparatus to collect oxygen by displacement of water. Use a 500-cc. bottle for this purpose. Heat the mixture until the potassium chlorate is entirely decomposed — *i.e.*, no more oxygen is evolved — and disconnect the test tube from the delivery tube at once to prevent it from drawing in water as it cools. Raise or lower the bottle of oxygen until the levels of the water inside and outside are exactly the same. Now slip a glass plate under the mouth of the bottle, hold it tightly against the bottle and remove the bottle and contents to an upright position on the desk. The volume of the oxygen may now be measured indirectly by measuring a corresponding volume of water. Remove the glass plate and, by pouring water from a graduated cylinder, carefully note the amount

required to fill the bottle. Record this measurement as the volume of oxygen collected at room conditions. Take the temperature of the water, read the barometer and correct the pressure for the vapor pressure of water at the observed temperature (table in back of book). From this data calculate the volume which the oxygen would occupy at standard conditions — 0° Centigrade and 760 mm. pressure.

When the tube is cold weigh it again. What does the loss in weight represent? What is the per cent of oxygen in potassium chlorate according to your data? According to the formula?

From the corrected volume and the weight of the oxygen calculate the weight of a liter of oxygen under standard conditions. Compare your result with the accepted weight for this gas and calculate the percentage error. List the probable sources of error in your determination. Is the result affected by the fact that the test tube and delivery tube were full of air at the beginning of the experiment and full of oxygen at the end? Why is it necessary to correct for the vapor pressure of water? Would the observed volume of oxygen have been greater or smaller if it had been collected over dry mercury?

Calculate the weight of potassium chlorate required to give a liter of oxygen at standard conditions.

State the law illustrated by the fact that, with careful work, different experimenters obtain the same result for the percentage of oxygen in various samples of potassium chlorate.

Each observation should be recorded directly in your notebook as soon as made. A table such as the following is suggested.

TABLE OF DATA

Weighing 1.	Weight of tube and MnO_2	g.
Weighing 2.	Weight of tube and MnO_2 and KClO_3 . . .	g.
	Weight of KClO_3 used	g.
Weighing 3.	Weight of tube and contents after heating	g.
	Weight of oxygen expelled	g.
	Volume of oxygen collected	cc.
	Temperature of the water	$^{\circ}\text{C}$.
	Barometric pressure	mm.
	Corrected pressure of the oxygen	mm.
	Corrected volume of the oxygen (S. C.) . . .	cc.
	(Indicate calculation)	

EXERCISE 12

METHODS OF PREPARING HYDROGEN

Textbook: 75-81

(a) **Action of Water on Metals.** — (*Caution:* Sodium is kept under kerosene and must not come in contact with moist fingers. Handle it with forceps.) Pour 10 cc. of water into a beaker and drop in a piece of sodium (storeroom) about the size of wheat grain (?). Float a piece of filter paper on the water and place on it another small piece of sodium (?). Why is the kindling temperature reached in this case and not before? Test the solution in the beaker with red litmus (?). Rub some of the solution between thumb and forefinger (?). Repeat this and the litmus test with some sodium hydroxide from the shelf. Write the equation for the action of sodium on water.

Recall the lecture experiment in which steam was passed over hot iron (equation). Compare this and the above reaction with regard to the type of products and with regard to the amount of hydrogen obtained from 36 g. of water. How else may hydrogen be obtained from water?

(b) **Action of Acids on Metals.** — Place a piece of zinc in each of three test tubes and cover one with dilute hydrochloric, one with dilute nitric and one with dilute sulfuric acid. Test the evolved gas with a lighted match or burning splinter (?). A slight explosion in the test tube indicates the presence of hydrogen. What salt is left in solution in each case?

In different test tubes try the action of hydrochloric acid on bits of the following metals: aluminum, tin, magnesium, zinc, iron, copper. If dilute hydrochloric acid has no effect pour it off and add the concentrated acid. Arrange the metals in the order of the activity with which they appear to displace hydrogen.

Why should not sodium be used with an acid to generate hydrogen? What metal might be used to make a container for hydrochloric acid? (*See Electromotive Series.*)

EXERCISE 13

PREPARATION AND PROPERTIES OF HYDROGEN

Textbook: 86-91

(a) **Preparation.** — (*Caution:* Hydrogen is explosive when mixed with air. Do not bring a flame near the hydrogen generator.) Fit a flask with a rubber stopper bearing a thistle tube and delivery tube as shown in Figure 12. Place 10 g. of zinc in the flask and add enough water to cover the end of the thistle tube. Pour 20 cc. of dilute sulfuric acid down the thistle tube and add more when needed. Collect the gas in wide-mouthed bottles by displacement of water. If the action is slow, add through the thistle tube a few cc. of copper sulfate solution. The apparent catalysis in this case is caused by metallic copper depositing on the zinc and forming with it an electric couple. The first bottle of gas collected will be a mixture of air and hydrogen and may be discarded or exploded by bringing its mouth to a flame.

(b) **Combustion.** — Collect a bottle of pure hydrogen and, while holding it mouth downward, apply a lighted candle or match attached to a wire or piece of glass tubing (?). Thrust the candle further up into the hydrogen (?). Withdraw it slowly (?). State your conclusion as to the ability of hydrogen to burn and to support combustion. What is the color of the hydrogen flame? Does it produce much heat? What product do you see formed in the bottle? Equation. *Hydrogen + Oxygen*

(c) **Lightness.** — Hold a bottle of air mouth downward and pour a bottle of hydrogen **upward** into it. Now test both bottles with a flame (?). From the weights of a liter of the gases (text-book) indicate the relative weights of air and hydrogen. What use is made of this property of hydrogen? What is the objection to hydrogen for this use? What substitute is there?

(d) **Explosiveness.** — Fill a wide-mouthed bottle about a third full of hydrogen, drop the remainder of the water out allowing air to enter and wait a moment for the gases to mix. Apply a flame to this mixture and compare the result with that obtained in (b).

(e) **Diffusion.** — Obtain a cylinder of unglazed porcelain and fit it with a rubber stopper bearing a glass tube about 25 cm.

long. Let the end of this tube dip into a beaker of water and bring a bottle of hydrogen down over the porous cylinder. If the mouth of the bottle is too small pour the hydrogen **upward**

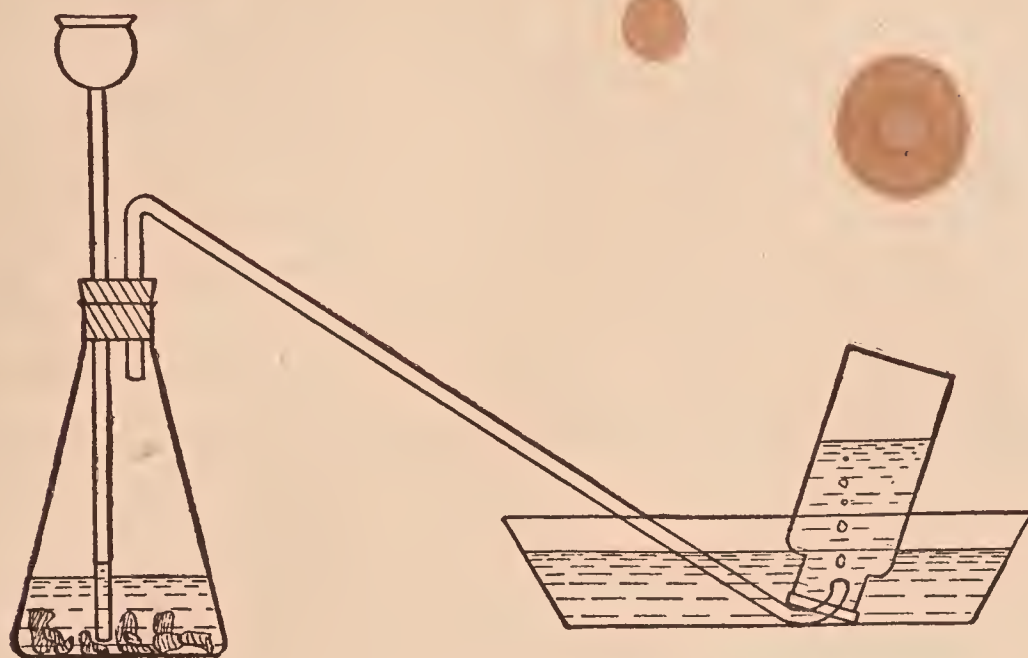


FIG. 12

into a large beaker placed over the cylinder (?). After a minute remove the vessel of hydrogen and watch the level of water inside the tube (?). In your opinion, why can hydrogen pass through the pores of the unglazed porcelain faster than can air?

(f) **Other Product.** — Filter about ~~2 cc.~~ *add 10 cc. of water* of the liquid in the flask into a watch glass and evaporate this on a water bath. Describe the residue. To what class of substance does it belong? Write the chemical equation to show how it was formed. *in work p 239*

(g) **Problem.** — Calculate the weight of hydrogen possibly liberated by the weight of the zinc you used if it had all dissolved. Calculate the volume of this hydrogen if a liter weighs 0.089 g. at standard conditions. About what per cent of this volume did you actually utilize in this exercise?

EXERCISE 14

HYDROGEN AS A REDUCING AGENT

Textbook: 77-80

Pour off the liquid from the hydrogen generator used in the previous exercise and if sufficient zinc is left use it for this ex-

ercise, providing more if necessary. Prepare a right-angled delivery tube A, set up the apparatus as shown in Figure 13 and prepare hydrogen as in Exercise 13 (a).

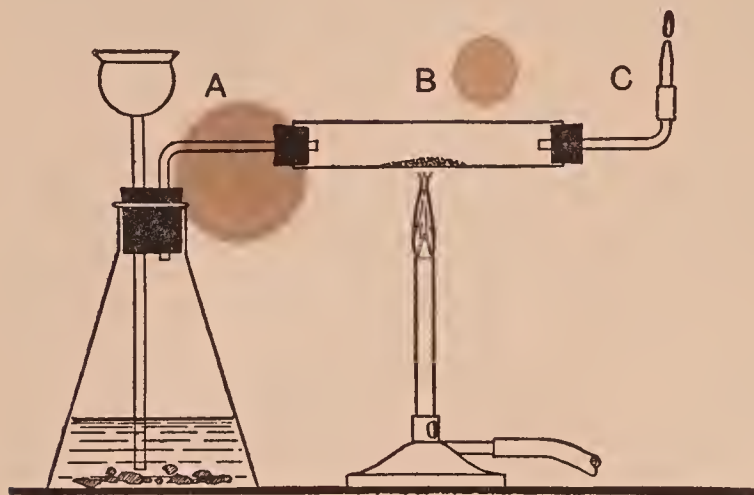


FIG. 13

(a) **Reduction of Copper Oxide.** — Place about 0.5 g. of copper oxide in the middle of the ignition tube B. When the hydrogen generator has been working long enough to displace air from the apparatus, wrap a towel

around it and light the glass tip C by collecting a test tube full of pure hydrogen over it and using this tube of hydrogen as a torch. (Recall the method as shown in the lecture or have your instructor demonstrate.) **Never light a hydrogen generator by any other method.** Hold a cold dry beaker over the hydrogen flame and note the deposit (?). Hold a piece of iron wire in the flame (?).

Heat the copper oxide with the Bunsen burner; notice the effect upon the hydrogen flame (?), any deposit beyond the copper oxide (?) and any change in the appearance of the copper oxide (?). Explain the results, giving the equation.

(b) **Reduction of Iron Oxide.** — Repeat the experiment, using magnetic oxide of iron in place of copper oxide and make observations as above. Recall the action of steam on iron, Exercise 12 (a), and explain. What kind of reaction is this? What determines its direction? Is the reduction of copper oxide reversible? Can copper ever displace hydrogen? Recall 12 (b).

EXERCISE 15

WEIGHT OF METAL TO DISPLACE A GRAM ATOM OF HYDROGEN

Quantitative — Formal Report Required

Obtain a large test tube, fit it with a two-holed stopper and set up the apparatus as illustrated in Figure 14. Weigh ac-

curately a strip of magnesium ribbon about 6 cm. long. Place this strip in the test tube and fill the whole apparatus with water by pouring it into the funnel and regulating the flow by means of the pinch-cock. Be careful to drive out any air bubbles and make sure there are no leaks in the system. Keep some liquid in the funnel throughout the experiment. Fill the graduated cylinder with water and invert it over the delivery tube in a beaker of water.

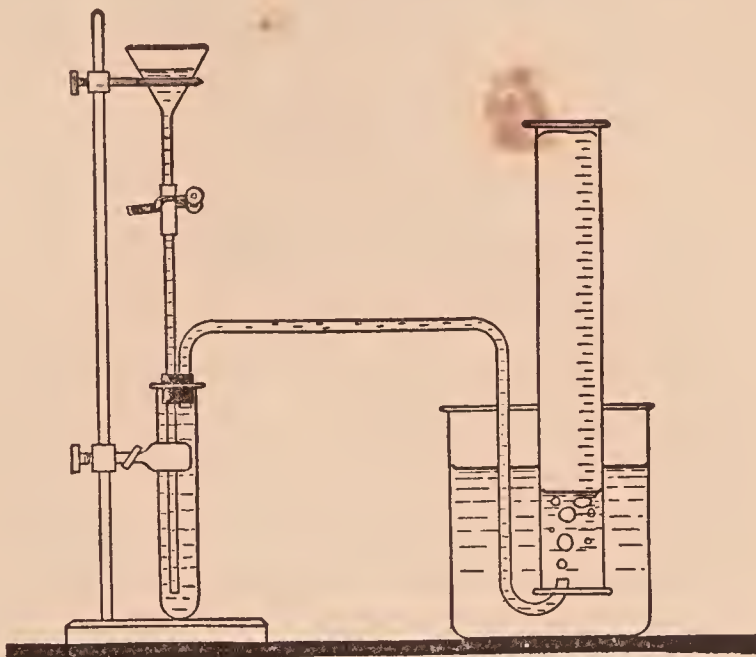


FIG. 14

Pour 10 cc. of concentrated hydrochloric acid into the water in the funnel and allow it to flow into the tube until the magnesium has dissolved. When this has occurred, drive all the gas from the system into the graduated cylinder by pouring water into the funnel and allowing it to flow through the tubes.

Bring the gas in the cylinder to atmospheric pressure (?) and read the volume. Calculate the volume the gas would occupy at standard conditions. Should you make a correction for vapor pressure of water? From the weight of a liter of hydrogen at standard conditions calculate the weight of hydrogen you obtained. Arrange your data in a table as in Exercise 11.

From your data calculate the weight of magnesium required to displace a **gram atom** of hydrogen. What is this weight of magnesium called? Compare this with the theoretical weight (equation) and calculate your percentage error.

What is the equivalent weight of aluminum? Of oxygen? Of sulfuric acid? Compare the amounts of hydrogen displaced by gram atoms of sodium, magnesium, and aluminum. What is the relation between the equivalent weight and the atomic weight of an element?

Answer - 12.16 g

hydrogen gas = 7.44 - 18.0

EXERCISE 16

WATER — BOILING AND FREEZING POINTS —
PURIFICATION *by method of distillation**Textbook: 108-109; 114-118*

(a) **Effect of Solutes.** — Place 20 cc. of water in a 100-cc. beaker or flask and heat this until it boils. Regulate the flame to keep it boiling gently. Immerse the bulb of a thermometer in the boiling water and read the temperature finally reached (?). Remove the thermometer and saturate the boiling water with common salt (about 8 g. needed). When this solution is boiling steadily read the maximum temperature reached. How does the use of salt affect the time required to cook eggs, vegetables, etc., in water? What external condition affects the boiling point of a liquid? What is the vapor pressure of water at 100°C.? What is the effect of a dissolved substance upon the vapor pressure of the solvent?

In the same manner NaCl and other soluble substances lower the freezing point of water. If any crushed ice or snow is available this statement should be verified. Can you think of any

practical application of this fact? Explain the use of "anti-freeze" mixtures in automobile radiators in winter.

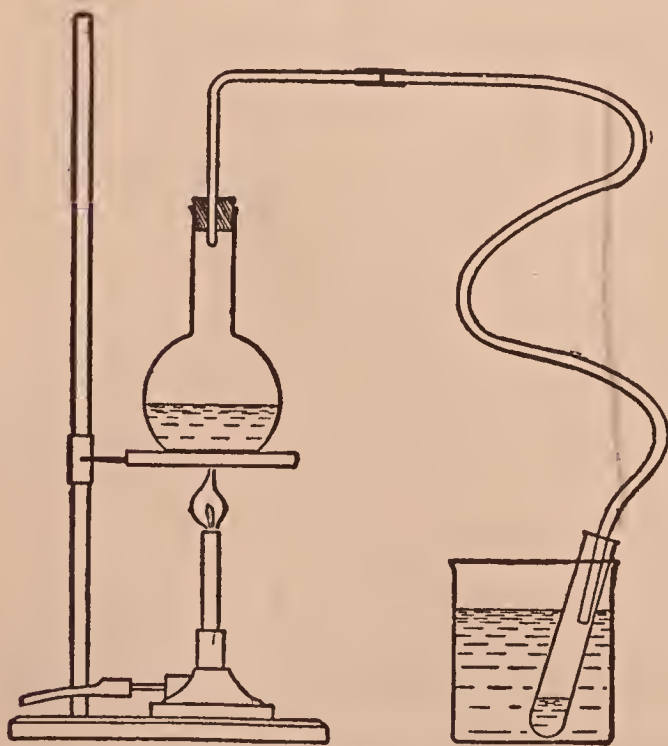


FIG. 15

(b) **Preparation of Distilled Water.** — Fit a small flask with a one-hole stopper and prepare a condensing tube as shown in Figure 15. The condenser may be made in spiral form if a bit of skill at glass working is possessed. To 40 cc. of water add 2 cc. of copper sulfate solution and a like

amount of sodium chloride solution. Mix well and test three 2-cc. portions with NH_4OH (?), with BaCl_2 solution (?), and with AgNO_3 solution (?). These tests show respectively the

presence of copper, a sulfate, and a chloride. Put the remainder of the solution in the flask. Discard the first 5 cc. of the distillate and collect 10 cc. more. Make the above three tests on small portions of the distillate (?).

D Can all substances be removed by distillation? Explain your answer.

EXERCISE 17

SOME PROPERTIES OF WATER — SOLUTIONS

Textbook: 119; 140-141

(a) **Combination with Anhydrides.** — Moisten a small piece of quicklime (calcium oxide) with water and test with litmus paper (?). Equation. Recall from Exercise 9 the union of water with other basic and acidic oxides.

(b) **As a Medium of Chemical Action.** — Pulverize a gram of lead nitrate in a mortar and heat the powder in a porcelain dish to dry it. Thoroughly clean the mortar and pestle and pulverize a gram of potassium iodide. Heat this powder to dryness in another dish or crucible. When both substances are cool mix them in one dish (?). Now add a drop of water to the mixture of dry salts (?). Add more water and write the equation for the reaction. Why is water necessary for this chemical action?

(c) **Temperature Effect of Solution.** — Draw a beaker of water from the tap and measure its temperature with a thermometer. In a test tube place 2 g. of calcium chloride and dissolve it by shaking with the least amount of water. Note the temperature of the calcium chloride solution (?). In the same manner dissolve 2 g. of ammonium chloride and take the temperature of the solution (?). Compare the temperatures of the solutions with that of the water used. This temperature effect is due to the **Heat of Solution** which may be positive or negative for different substances.

EXERCISE 18

HYDROGEN PEROXIDE

Textbook: 121-125

(a) **Preparation.** — Pour into a small beaker 90 cc. of water and 10 cc. of dilute sulfuric acid. To this add, in small portions, about 2 g. of sodium peroxide, stirring vigorously. The solution should now be acid (test?), if not, add more sulfuric acid diluted as above. Write the equation. Recall the action of water alone on sodium peroxide — Exercise 6 (c).

(b) **Test.** — To 5 cc. of this hydrogen peroxide solution add about 3 cc. of ether, 2 or 3 drops of potassium dichromate solution and a few drops of dilute sulfuric acid. Place your thumb over the mouth of the test tube and shake it. The color (?) in the ether layer is used as a test for hydrogen peroxide or, conversely, for a dichromate.

(c) **Stability.** — To 10 cc. of the solution add a small amount of manganese dioxide (?). Test the evolved gas with a glowing splinter (?). Is hydrogen peroxide a stable compound? Write the equation for its decomposition. What conditions accelerate its decomposition? Why is hydrogen peroxide usually sold in brown bottles?

(d) **As a Reducing Agent.** — Dilute a few drops of potassium permanganate solution with 5 cc. of water. Add this solution in small portions with shaking to 10 cc. of hydrogen peroxide solution. What gas is evolved? Refer to the textbook for the equation. What is the rôle of potassium permanganate in this reaction?

(e) **As an Oxidizing Agent.** — Dilute 1 cc. of lead nitrate solution to 10 cc. and add some hydrogen sulfide solution. Shake the tube to suspend the black precipitate (?), pour some of this into another tube and add hydrogen peroxide. What happens to the black precipitate? Write the equations.

(f) **Evaluation of a Solution of Hydrogen Peroxide.** — The strength of a given solution of hydrogen peroxide may be determined by two methods:

(1) The volume of oxygen gas which can be obtained from a definite volume of the hydrogen peroxide solution may be measured. For example, the 3 per cent commercial product is

commonly referred to as a 10-volume solution because one volume of this solution will liberate ten volumes of oxygen gas. If the oxygen evolved in part (c) of this exercise had been measured, this method would have been illustrated.

(2) If a measured quantity of a solution of potassium permanganate of known strength is added to a definite volume of hydrogen peroxide solution until a pink color just persists in the solution, the strength of the hydrogen peroxide may be calculated from the quantity of the permanganate solution used.

The solution of potassium permanganate on the shelf contains 15.8 g. of the salt per liter of solution. Dilute 20 cc. of this solution to 100 cc. and calculate the strength of this diluted solution. Obtain a burette (see Fig. 20) and fill the burette to the zero mark with the permanganate solution you have prepared, first making sure the air bubbles have been displaced from the rubber connection and tip by bending the tip upward and allowing a little of the solution to flow.

Pour exactly 10 cc. of your hydrogen peroxide solution into a beaker, add about 3 cc. of dilute sulfuric acid diluted with about 20 cc. of distilled water, and carefully add some of the permanganate solution from the burette. Stir the solution in the beaker constantly and run the permanganate solution into this until a faint pink color persists after stirring. The last drop of permanganate solution added should cause this change in color. Record the volume of permanganate solution used and check this titration with another 10-cc. portion of the hydrogen peroxide solution.

From your data calculate the weight of potassium permanganate which was reduced by 10 cc. of the hydrogen peroxide solution, and from this obtain the weight of hydrogen peroxide in 10 cc. of your solution and in a liter of such solution. What is the percentage of hydrogen peroxide in the solution? What weight of hydrogen peroxide should have been produced from 2 g. of sodium peroxide? If this quantity had been produced, what would be the percentage of hydrogen peroxide in your solution?

EXERCISE 19

THE LAW OF MULTIPLE PROPORTIONS

Textbook: 125-126

Quantitative

A. Refer to Exercise 11 and repeat exactly the directions for weighing and heating the materials, except substitute potassium perchlorate for potassium chlorate and do not collect the oxygen which is evolved.

Arrange your data from this experiment exactly as you did the weight data in Exercise 11. Calculate from the two sets of data the weight of oxygen which is combined with one gram of potassium chloride and with one gram molecule of potassium chloride in potassium chlorate and in potassium perchlorate.

Show from your results that a simple, whole-number ratio exists between the weights of oxygen combined with a fixed weight of potassium chloride in the two compounds.

State the law exemplified by this experiment. Recall or obtain from your textbook the formulas for five oxides of nitrogen and show clearly, by means of atomic weights, how these five compounds illustrate the law just stated.

B.* In a dry hard-glass test tube place about one gram of cupric bromide and weigh the tube and contents accurately. Clamp the tube in a position slightly inclined to the horizontal and fit it with a one-hole stopper (either cork or rubber) bearing a glass tube which dips into a flask containing a little water. The end of the tube should not dip below the surface of the water, but should end about one inch above it. Heat the cupric bromide in the tube, gently at first, then more strongly, until bromine fumes are no longer evolved. During this operation it is necessary to warm the upper portion of the tube somewhat, in order to prevent the condensation of bromine. When the tube has cooled, remove the stopper. Tilt the tube so that any bromine fumes which remain in it may escape, and weigh the tube and its contents. Now pour 2 cc. of concentrated nitric acid into the test tube, replace the stopper and delivery tube,

* This experiment was contributed by Dr. John C. Bailar, Jr., of the University of Illinois. Cf. *Jour. Chem. Educ.* 6, 1760 (1929).

and boil the liquid gently until all of the acid has evaporated, and the residue is dry and black. Replace the one-holed stopper by one having two holes; through one hole should pass a tube reaching a little more than half way to the bottom of the test tube, through the other a tube which extends not more than half an inch beyond the inner surface of the stopper. Connect one of these to a hydrogen generator; when a test shows that all air has been expelled from the apparatus heat the material in the test tube until it is completely reduced to metallic copper. Allow the tube to cool, while the stream of hydrogen is still passing through it, and weigh it again. The data may be recorded in the following manner:

1. Weight of test tube and cupric bromide.....
2. Weight of test tube and cuprous bromide.....
3. Weight of test tube and copper.....
4. Weight of bromine in the cupric bromide.....
5. Weight of bromine in the cuprous bromide.....
6. Ratio of the two amounts of bromine (4 divided by 5).....

State the chemical change which occurs at each step in this experiment, writing the equation if possible. Explain clearly how your results exemplify the Law of Multiple Proportions.

EXERCISE 20

SOLUBILITY — SUPERSATURATED SOLUTION

Textbook: 135-139

(a) **Influence of Temperature.** — To 5 cc. of water in a test tube add ~~10~~ cc. of lead nitrate solution and mix by shaking. Prepare a similar dilute solution of sodium chloride and add half of each solution to a third test tube (?). Heat the remaining halves of the solutions to boiling and mix them (?). Cool this mixture by immersing the test tube in a beaker of water (?). Heat the other tube containing the precipitate (?). What is the precipitate? Equation. What can you say of the solubility of this substance? Recall the relation of temperature to solubility of NaCl and KNO₃ studied in Exercise 4.

(b) **Influence of Size of Particles.** — Using two small squares of paper of exactly equal size counterbalance on the pans of a

chemical balance a *single crystal* (not too large) of copper sulfate or potassium dichromate with some of the same substance previously powdered in a mortar. Place the two samples in test tubes, add equal amounts of water (10 or 15 cc.), shake the two tubes equally and measure the time required for each sample to completely dissolve. State the factors which influence rate of solution.

(c) **Supersaturation.** — Weigh out 5 g. of Glauber's salt ($\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$) and place it in a test tube with 4 cc. of water. Warm and shake the tube until all the salt is dissolved. Set the test tube in a beaker of cold water or cool it under the tap. If no crystals have appeared when the solution is cool, you have a supersaturated solution. Now drop into the solution a crystal of the salt (?). What kind of solution do you now have? How could you determine whether a solution is unsaturated, saturated or supersaturated at a given temperature?

EXERCISE 21

HYDRATES

Textbook: 120; 139-140

(a) **Test for Water of Hydration.** — Heat a ^{2 spoon} gram of each of the following crystals in dry test tubes and notice whether an appreciable amount of water is given off in each case: sodium chloride; copper sulfate; sodium phosphate; potassium dichromate. If any are found to be hydrates write their complete formulas. *NaCl, Na₂HPO₄ · 10H₂O*

(b) **Efflorescence and Deliquescence.** — Place a few granules of calcium chloride on a watch glass or glass plate, some clear crystals of Glauber's salt on another, and a clear crystal of copper sulfate on another. Set these away in your desk until the next laboratory period, then note and explain any changes. Define a deliquescent and efflorescent substance and illustrate from your experiment.

Put a gram of granulated calcium chloride in the bottom of a test tube, place a thin tuft of cotton over this, and on the cotton lay a clear crystal of copper sulfate. Close the tube tightly with a stopper and set away until the next period. Compare the appearance of the copper sulfate with that of the sample left on a watch glass. Explain.

(c) **Hydration.** — Mix 10 g. of plaster of Paris $((\text{CaSO}_4)_2 \cdot \text{H}_2\text{O})$ and 10 cc. of water in a small beaker, stirring the mixture until a thin paste is formed. Pour this into a filter paper fitted in a funnel and allow the plaster to “set.” After an hour or more examine the product. It is gypsum (formula?); write the equation. (See Textbook: p. 610.)

EXERCISE 22

FORMULA OF A HYDRATE

Quantitative

Heat a clean porcelain crucible to redness on a clay triangle, supported by the ring stand. Allow the crucible to cool and weigh it accurately. Put into it about one gram of gypsum and weigh it again. Heat the crucible and gypsum as before to redness for 5 minutes, allow it to cool and weigh the crucible and contents again. From the amount of gypsum used and the loss in weight, calculate the per cent water of hydration in the original compound. Tabulate your data.

Gypsum is a hydrate of calcium sulfate. Using the atomic weights of the elements involved and your own data, calculate the correct formula of the hydrate. The following proportion is suggested for the solution of this problem:

$$\frac{\text{Molecular weight of CaSO}_4}{x \text{ times mol. wt. of H}_2\text{O}} = \frac{\text{Weight of CaSO}_4 \text{ left}}{\text{Loss in weight.}}$$

Solve for x to obtain the number of molecules of water in a molecule of the hydrate. The result should be very nearly a whole number. Any variation is due to impurity of the original material or to errors in your work. Now write the complete formula of gypsum and calculate the theoretical per cent of water of hydration. Compare this with your experimental result and determine your percentage error.

Other hydrates, such as borax and crystallized barium chloride, may be substituted for gypsum in this experiment. $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ is likely to give incorrect results due to partial decomposition of the sulfate into CuO and SO_3 if the temperature becomes too high.

chemical balance a *single crystal* (not too large) of copper sulfate or potassium dichromate with some of the same substance previously powdered in a mortar. Place the two samples in test tubes, add equal amounts of water (10 or 15 cc.), shake the two tubes equally and measure the time required for each sample to completely dissolve. State the factors which influence rate of solution.

(c) **Supersaturation.** — Weigh out 5 g. of Glauber's salt ($\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$) and place it in a test tube with 4 cc. of water. Warm and shake the tube until all the salt is dissolved. Set the test tube in a beaker of cold water or cool it under the tap. If no crystals have appeared when the solution is cool, you have a supersaturated solution. Now drop into the solution a crystal of the salt (?). What kind of solution do you now have? How could you determine whether a solution is unsaturated, saturated or supersaturated at a given temperature?

EXERCISE 21

HYDRATES

Textbook: 120; 139–140

(a) **Test for Water of Hydration.** — Heat a ^{upron} gram of each of the following crystals in **dry** test tubes and notice whether an appreciable amount of water is given off in each case: sodium chloride; copper sulfate; sodium phosphate; potassium dichromate. If any are found to be hydrates write their complete formulas. *Na₂HPO₄ · 12H₂O*

(b) **Efflorescence and Deliquescence.** — Place a few granules of calcium chloride on a watch glass or glass plate, some clear crystals of Glauber's salt on another, and a clear crystal of copper sulfate on another. Set these away in your desk until the next laboratory period, then note and explain any changes. Define a deliquescent and efflorescent substance and illustrate from your experiment.

Put a gram of granulated calcium chloride in the bottom of a test tube, place a thin tuft of cotton over this, and on the cotton lay a clear crystal of copper sulfate. Close the tube tightly with a stopper and set away until the next period. Compare the appearance of the copper sulfate with that of the sample left on a watch glass. Explain.

(c) **Hydration.** — Mix 10 g. of plaster of Paris $((\text{CaSO}_4)_2 \cdot \text{H}_2\text{O})$ and 10 cc. of water in a small beaker, stirring the mixture until a thin paste is formed. Pour this into a filter paper fitted in a funnel and allow the plaster to “set.” After an hour or more examine the product. It is gypsum (formula?); write the equation. (See Textbook: p. 610.)

EXERCISE 22

FORMULA OF A HYDRATE

Quantitative

Heat a clean porcelain crucible to redness on a clay triangle, supported by the ring stand. Allow the crucible to cool and weigh it accurately. Put into it about one gram of gypsum and weigh it again. Heat the crucible and gypsum as before to redness for 5 minutes, allow it to cool and weigh the crucible and contents again. From the amount of gypsum used and the loss in weight, calculate the per cent water of hydration in the original compound. Tabulate your data.

Gypsum is a hydrate of calcium sulfate. Using the atomic weights of the elements involved and your own data, calculate the correct formula of the hydrate. The following proportion is suggested for the solution of this problem:

$$\frac{\text{Molecular weight of CaSO}_4}{x \text{ times mol. wt. of H}_2\text{O}} = \frac{\text{Weight of CaSO}_4 \text{ left}}{\text{Loss in weight.}}$$

Solve for x to obtain the number of molecules of water in a molecule of the hydrate. The result should be very nearly a whole number. Any variation is due to impurity of the original material or to errors in your work. Now write the complete formula of gypsum and calculate the theoretical per cent of water of hydration. Compare this with your experimental result and determine your percentage error.

Other hydrates, such as borax and crystallized barium chloride, may be substituted for gypsum in this experiment. $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ is likely to give incorrect results due to partial decomposition of the sulfate into CuO and SO_3 if the temperature becomes too high.

EXERCISE 23

COLLOIDAL SOLUTIONS

Textbook: 147-155

Colloidal dispersions ("solutions") are systems in which the dispersed ("dissolved") phase consists of particles considerably larger, in general, than the molecules in a true solution and somewhat smaller than the particles in a suspension which can be separated by filtration or settling. It is obvious, therefore, that colloidal dispersions may be prepared by: (1) *Dispersion methods* which involve breaking up larger aggregates into particles of colloidal dimensions and (2) *Condensation methods* by which substances in true solution are caused to unite and form colloidal particles.

Colloidal particles do not ordinarily settle out, are not removed by filtration, are not visible in a microscope, but their presence may be detected by an ultramicroscope due to the Tyndall effect. Colloidal particles are electrically charged and may be coagulated by the addition of an electrolyte. That ion of the electrolyte which bears the charge opposite to that of the colloidal particles causes this coagulation, and the effectiveness of such an ion increases with its charge or valence.

(a) **Sulfur.** — Place a few milligrams of powdered sulfur in a test tube, add about 2 cc. of alcohol and shake the tube at intervals for five minutes to dissolve the sulfur. It is not necessary that all the sulfur be dissolved. Now pour the alcoholic solution of sulfur into about 15 cc. of water in a test tube (?). Keep all of the colloidal solutions which you prepare, exhibit them to your instructor at the end of this exercise and preserve them in corked test tubes for at least several days in order to observe their stabilities.

Prepare a sample of colloidal sulfur by the interaction of a dilute solution of sodium thiosulfate with very dilute sulfuric acid. If the sulfur obtained by this method tends to settle out, the solutions should be diluted still further before mixing.

Colloidal sulfur may also be produced by oxidizing a solution of hydrogen sulfide by simply exposing it to the air or by adding to it a small quantity of acidified potassium permanganate solution or other oxidizing agent.

(b) **Silver.** — Add ammonium hydroxide solution to a small volume of dilute silver nitrate solution until the precipitate which first forms is redissolved (?). Now add to this a small quantity of a reducing agent such as glucose solution, alcohol, or formaldehyde. Warm this preparation until a faint but definite color develops (?).

(c) **Silver Iodide.** — Add 7 cc. of 0.1 normal solution of silver nitrate to 20 cc. of 0.05 normal potassium iodide solution (?). Consult your instructor with regard to the preparation of these solutions from the solutions on the shelf if the latter have a higher concentration.

Repeat this preparation after first adding 1 cc. of a one per cent solution of gelatin to each portion of the reactant solutions.

Prepare a saturated solution of sodium chloride by shaking 10 g. of the salt with 25 cc. of water in a small flask until no more of the salt will dissolve. Determine the minimum volume of this solution which must be added to 10 cc. of your first preparation of silver iodide in order to produce an evident effect of coagulation (?). Add this quantity of the sodium chloride solution to 10 cc. of the silver iodide "solution" containing gelatin (?). Explain the protective effect of the gelatin. Mention several other protective colloids which might have been used. State an example of the use of a protective colloid in a commercial product.

(d) **Hydrated Iron Oxide.** — Heat 50 cc. of water to the boiling point; while it is gently boiling add 5 drops of a solution of ferric chloride and continue to boil the solution for several minutes (?). Add 5 drops of the ferric chloride solution to 50 cc. of cold water and compare the intensity of color in the two preparations. What reaction has occurred in the hot solution? If the cold solution is permitted to stand, the color will gradually deepen somewhat, showing that the same reaction is occurring in this solution but more slowly. Explain how *dialysis* would aid in carrying this reaction toward completion in the cold solution.

EXERCISE 24

GELS

Textbook: 153-154

(a) **Soap Gels.** — Place one gram of soap in each of two small beakers. To one of these add 19 cc. of water and to the other add 49 cc. of water. Warm each preparation (do not boil) until the soap is uniformly dispersed. Permit both solutions to cool to room temperature and let them stand undisturbed for an hour or longer. Do either or both of the solutions gel? What is the percentage concentration of soap in each solution? Is a dispersion of soap in water a true solution or a colloidal solution?

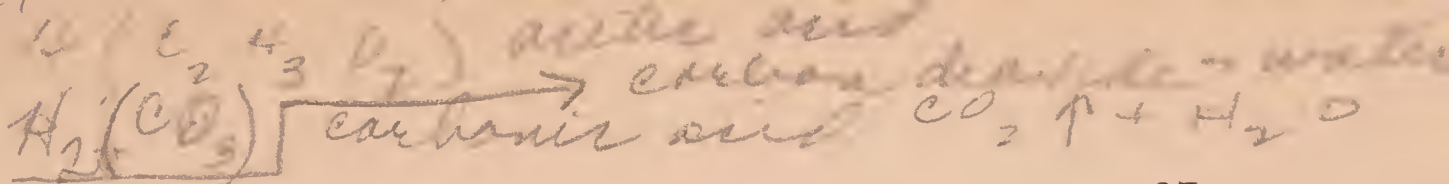
(b) **Silica Gels.** — Dilute 25 cc. of water glass (egg-preservative grade may be used) with 75 cc. of water and mix the solution thoroughly. To 10 cc. of this solution add a drop of phenolphthalein solution and determine approximately the quantity of dilute acetic acid (about normal concentration should be used) required to decolorize this solution. Add a slightly greater quantity of the acetic acid, all at once, to another 10-cc. portion of the water-glass solution, mix quickly and let this preparation stand until it gels. If the gel forms too quickly so that the solutions can not be mixed uniformly, the solutions should be diluted still further before mixing. By a few trials the proper concentrations to produce a clear, uniform gel can be determined.

What is the composition of water glass (Textbook: p. 426)? What reaction occurs when acetic acid is added to water glass? Equation.

Heat a piece of the silica gel in an evaporating dish until no further change occurs (?). What is the final product?

Warm the soap gel prepared in (a) but do not evaporate it to dryness (?). Gels may be classified with respect to the effect of heat as *reversible* and *non-reversible*. Upon this basis classify the gels you have prepared. Mention several other gel-forming substances in addition to those used in this exercise.

(c) **"Silicate Garden."** — Place the remainder of the water-glass solution prepared in (b) in a small beaker — at least 50 cc. of the solution should be used for this experiment — and drop



into this solution several small crystals of any of the following salts: cobalt nitrate, ferric chloride, nickel nitrate, copper nitrate or chloride, zinc nitrate or chloride. Permit this preparation to stand for at least an hour. Observe and explain, in terms of osmotic pressure, the growths obtained in your "silicate garden" (Textbook: p. 145).

How does a gel differ from a gelatinous precipitate such as is obtained when ammonium hydroxide is added to a solution of an aluminum or ferric salt?

EXERCISE 25

DOUBLE DECOMPOSITION OR METATHESIS

Textbook: 141

When two compounds react in solution without any valence changes occurring, two other compounds may be completely produced if:

- One of the products is insoluble and precipitates.
- One of the products is a gas and escapes.
- One of the products is ~~slightly ionized~~ — e.g., H_2O .

Mix the following solutions in pairs, using about 5 cc. of the first solution and adding the second until no further change occurs:

- Sodium chloride and silver nitrate. — *cloudy white precipitate*
- Barium chloride and dilute sulfuric acid. — *white precipitate*
- Sodium carbonate and acetic acid. — *effervescent gas*
- Copper nitrate and sodium hydroxide. — *blue precipitate*
- Ferric chloride and ammonium hydroxide. — *brown precipitate*
- Precipitate from (4) and hydrochloric acid. — *precipitate dissolves*
- Precipitate from (5) and nitric acid. — *dissolves*

Write the equations and indicate in each case the condition (a, b or c above) which caused the reaction to go to completion.

Complete the following type equations:

An acid plus a base gives —

An acid plus a salt gives —

A base plus a salt gives —

A salt plus a salt gives —

Illustrate each type with examples other than those given in the above experiment.

EXERCISE 26

PREPARATION AND PROPERTIES OF CHLORINE

Textbook: 174-191

(a) **Preparation.** — Set up a flask as in Figure 16 with a delivery tube extending down into a dry bottle. Cover the bottle with a piece of cardboard or heavy paper having a small hole through which the delivery tube passes. (The remainder of the apparatus shown in Figure 16 is not used in this exercise.)

Calculate the weight of manganese dioxide needed to prepare 2 l. of chlorine by the oxidation of hydrochloric acid. Place this weight of manganese dioxide in the flask and add 15 cc. of concentrated hydrochloric acid through the thistle tube. Be sure the end of the thistle tube dips into the acid.

Collect and cover with glass plates three bottles of chlorine, heating the generator if necessary, and then allow the chlorine to bubble into 50 cc. of water in a small flask to prepare "chlorine water." When this becomes colored fill the generator with water to stop the reaction.

(b) **Bleaching.** — In one bottle place a piece of wet, colored calico; a dry piece of the same material; wet pieces of paper bearing print, pencil and ink writing. Be careful to keep the dry cloth out of contact with the wet pieces. After a time observe and explain what has happened. Indicate by means of an equation the rôle of water in bleaching with chlorine.

(c) **Reaction with Antimony.** — Sift a little powdered antimony into a bottle of chlorine (?). Equation. Add water and note the formation of a white precipitate (SbOCl). Equation.

(d) **Reaction with Phosphorus.** — Ignite a small piece of phosphorus as in Exercise 9 (c) and lower it into a bottle of chlorine (?). Note whether liquid phosphorus trichloride or solid phosphorus pentachloride is formed and write the proper equation. Add some water to the product and test the solution with litmus paper. Explain, giving the equation. What element does chlorine most resemble with regard to its activity in combining with many other elements?

(e) **Chlorine Water.** — Test some of the chlorine solution with blue litmus, noting and explaining the two changes in color which occur. Leave a small piece of dark sponge in a tube of

chlorine water for some time (?). Try some chlorine water on the end of a glass rod as an "ink eradicator" (?). Would you recommend chlorine water to remove ink stains from dyed fabrics? What happens when chlorine water is exposed to sunlight?

EXERCISE 27

HYDROGEN CHLORIDE — HYDROCHLORIC ACID

Textbook: 193-207

(a) **Preparation — Physical Properties.** — To a gram of common salt in a dry test tube add 0.5 cc. of concentrated sulfuric acid. Taking care not to breathe in the gas, note its odor and color. Blow your breath across the mouth of the tube (?). Test the gas with moist litmus paper (?). Bring a rod moistened with ammonium hydroxide into the tube (?). Invert the tube in a dish of water and explain the rise of water in the tube.

Write the equation for the reaction and name the products. What is the relation of this gas to hydrochloric acid in bottles on the desk?

(b) **Test for Chlorides.** — Add silver nitrate solution to a few drops of each of the following, placed in separate test tubes: dilute hydrochloric acid; sodium chloride solution; barium chloride solution. Describe the results and write the equations. Remove the product from one of the tubes to a filter paper and expose it for some time to the brightest light available (?). To the product in another of the tubes add ammonium hydroxide and shake (?), then acidify it with nitric acid (?). State how you could now test for chlorides in a water supply.

(c) **Reaction with Metals.** — Recall the action of hydrochloric acid on metals — Exercise 12(b) — and write one equation to exemplify this.

(d) **Reaction with Hydroxides.** — To 1 cc. of dilute hydrochloric acid add 5 cc. of water, mix and divide the solution in three test tubes. Add 2 drops of methyl orange to one, 2 drops of litmus to the second, and 2 drops of phenolphthalein to the third. Record the colors. Carefully add ammonium hydroxide to the first, mixing well, until the color changes (?); likewise add limewater to the second (?), and sodium hydroxide to the third (?). Tabulate the colors of the three indicators in acid

and basic solutions. Write the reaction that occurred in each tube. What is this type of reaction called?

Add just enough dilute hydrochloric acid to make the solution containing phenolphthalein colorless and evaporate some of it to dryness in a porcelain dish. Taste the product (?).

(e) **Reaction with Oxides.** — Place about 0.25 g. of zinc oxide in a test tube and add some water. Is it soluble? Add dilute hydrochloric acid and shake (?). Equation.

Repeat the experiment using quicklime. Evaporate the solution to dryness. What is the residue? Leave it exposed to the air of the laboratory for some time. What property does it show?

Place a few milligrams of ferric oxide in a test tube with 2 or 3 cc. of concentrated hydrochloric acid and warm until solution occurs. Pour half of this solution into another test tube and dilute it with about 20 cc. of water. Drop an iron nail into the other half of the solution, allow the reaction to proceed for some time, then dilute with about 20 cc. of water. Compare the colors of the two solutions, explain the difference and write the equations. Also write the equation for the action of hydrochloric acid upon iron.

Add some dilute hydrochloric acid to a few milligrams of copper oxide in a test tube and warm until solution occurs. Equation. Dilute half of this solution and note a gradual change in the tone of the color. Compare this with solutions of other copper salts on the shelf (?). Drop an iron nail into the other portion of the solution. Why does the reaction gradually decrease? Wash and examine the nail (?). Equation.

(f) **Reaction with Oxidizing Agents.** — In separate test tubes place 0.1 g. of the following: manganese dioxide, potassium permanganate, potassium chlorate, potassium dichromate. What property do these substances have in common? To each add 1 cc. of concentrated hydrochloric acid; note the color and odor (Care) of the evolved gas and test it with moist litmus paper and with starch-iodide paper. To prepare starch-iodide paper thoroughly moisten a pinch of starch with cold water, add 50 cc. of boiling water and 1 cc. of potassium iodide solution, stir well and dip strips of filter paper into this mixture. Chlorine liberates iodine which forms a blue compound with starch; an excess of chlorine subsequently destroys this color. Hence this may be used as an indirect test for chlorine gas.

Summarize the chemical properties of hydrochloric acid as follows:

HCl REACTING WITH:	PRODUCTS IN GENERAL:	TYPE OF REACTION:
Metals		
Basic Oxides		
Hydroxides (Bases)		
Oxidizing Agent		

EXERCISE 28

POTASSIUM HYPOCHLORITE

Textbook: 209-214

Calculate the volume of chlorine needed to react with 5 g. of potassium hydroxide, then calculate the weight of potassium permanganate and the volume of concentrated hydrochloric acid (specific gravity, 1.2, containing 40 per cent HCl) required to produce this volume of chlorine.

Arrange apparatus as illustrated in Figure 16. In the flask A place approximately one-half more than the calculated weight of potassium permanganate.

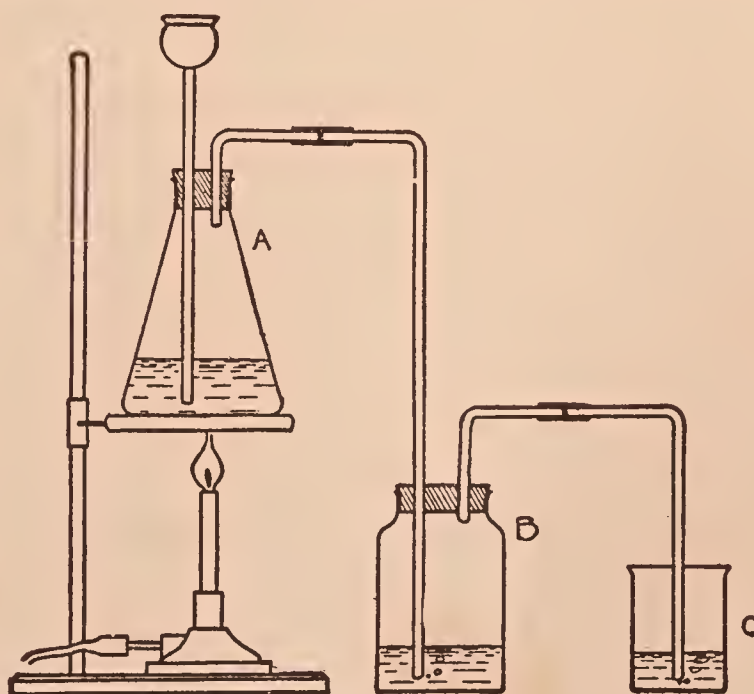


FIG. 16

Use a small bottle for B and add to it about 10 cc. of water. Have the tube from the chlorine generator dip into this; its purpose is to remove hydrogen chloride (very soluble) from the gas. Dissolve 5 g. of potassium hydroxide in 15 cc. of water, dilute 5 cc. of this solution to 20 cc. and reserve it in a test tube,

place the remainder of the concentrated solution in the small beaker C.

Pour one-half more than the calculated quantity of concentrated hydrochloric acid through the thistle tube and allow the chlorine produced to pass into the concentrated solution of potassium hydroxide until the solution no longer reacts alkaline to litmus paper but bleaches it. Note that the solution becomes warm. Write equations for the two reactions that have occurred in the solution. Set aside to crystallize and reserve the crystals for the next exercise.

Allow chlorine to bubble into the dilute solution of potassium hydroxide but do not saturate it (litmus test?). Equation. Acidify some of this solution and test its bleaching power with litmus paper, ink and colored cloth (?).

State the difference in the products obtained when chlorine is passed into dilute and into concentrated solutions of potassium hydroxide.

EXERCISE 29

POTASSIUM CHLORATE

Textbook: 216-218

Filter off the crystals prepared in the previous exercise and wash them twice with 2-cc. portions of water. Test some of the filtrate with silver nitrate solution (?). What product does this indicate? Dry the crystals between pieces of filter paper and try the following tests: 1. Dissolve a few crystals in water, acidify the solution with dilute HCl and compare its bleaching power with that of the hypochlorite solution (?). 2. Treat a few crystals with concentrated hydrochloric acid (?). 3. Heat the remainder of the crystals with a bit of manganese dioxide in a small test tube and test for oxygen (?).

EXERCISE 30

POTASSIUM PERCHLORATE

Textbook: 219-220

Heat 5 g. of potassium chlorate in a test tube until it melts and regulate the heat to keep it just above its melting point for

at least 15 minutes. At this temperature you may note a small amount of decomposition to form oxygen (equation?), but the principal reaction is the formation of the perchlorate (equation?).

When the tube is cool remove the solid, grind it in a mortar and transfer it to a beaker. Dissolve this mixture of salts by adding portions of water, heating it to boiling with stirring and pouring off the solution into another beaker. Repeat this process until the whole mass has been brought into solution. Allow the solution to cool and note the formation of crystals. What are they? What other salts are left in solution? What must be the relative solubility of the salts?

Carefully decant the mother liquor, bring the crystals on to a filter paper and wash them a few times with 2-cc. portions of water. Dry the crystals between filter papers; place a small quantity of them in a test tube; place a similar quantity of potassium chlorate in another tube and add concentrated hydrochloric acid to both (?). Which oxy-acid of chlorine is the most stable? Will an acidified solution of potassium perchlorate bleach litmus paper or cloth? Will potassium perchlorate give oxygen when heated? From the relative stabilities of the two salts would you expect the chlorate or the perchlorate to decompose at the lower temperature?

EXERCISE 31

BLEACHING POWDER

Textbook: 214-215

Place a small lump of quicklime in a 100-cc. beaker and carefully slake it by adding just enough water to cause the quicklime to crumble. Note the development of heat. Equation. Spread the slaked lime over the bottom of the beaker, generate some chlorine in a test tube (materials?) fitted with a stopper and right-angled delivery tube and allow the chlorine to pass into the beaker over the slaked lime. Note that absorption of the gas occurs and write the equation. The product $\text{CaCl}(\text{OCl})$, a "mixed salt," is the salt of what two acids? How would you name it to indicate this fact?

To a small portion of the bleaching powder add dilute sulfuric acid (?). Equation.

Mix some bleaching powder with 50 cc. of water, acidify this with a few drops of sulfuric acid and dip a piece of colored calico into the solution until a change is noted. Also try the action of this solution on news print and on a spot of fountain-pen ink on cloth (?). What is the actual bleaching agent in this solution? By means of an equation show the purpose of adding the sulfuric acid.

EXERCISE 32

IONIZATION

Textbook: 232-236

As indicated in Figure 17 allow the carbon rods attached to an electrical circuit to dip into a U-tube containing the following solutions separately:

- (a) Distilled water
- (b) Dilute hydrochloric acid
- (c) Sodium hydroxide solution
- (d) Sugar solution
- (e) Salt solution
- (f) Acetic acid solution
- (g) Alcohol solution
- (h) Ammonium hydroxide solution.

The U-tube and carbon electrodes must be rinsed with water after each trial before introducing the next solution.

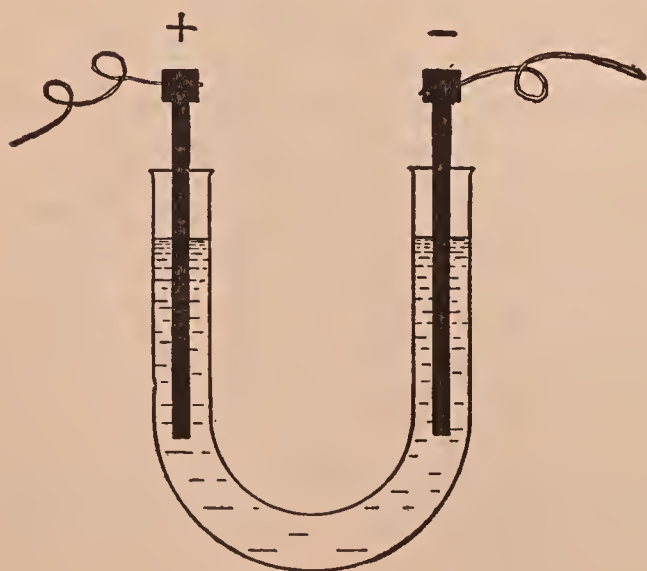


FIG. 17

If the electric light in the circuit glows when the switch is closed, conductance is shown, and the brightness of the light indicates the relative conductivity of the solution. What classes of substances conduct electricity in solution? Why? Indicate the ions, if any, that are present in each case. Define an electrolyte. Which substances

in the above solutions are electrolytes?

EXERCISE 33

IONIZATION AND CHEMICAL ACTION

Textbook: 232-238

(a) **H-Ion Concentration and Rate of Reaction.** — Prepare a series of mixtures of concentrated sulfuric acid and water in six test tubes as follows:

<i>Tube Number</i>	<i>Cc. Sulfuric Acid</i>	<i>Cc. Water</i>
1	10	0
2	8	2
3	6	4
4	4	6
5	2	8
6	0	10

In mixing the acid and water always pour the acid into the water and mix carefully. When the mixtures have cooled to room temperature, drop a piece of zinc into each of the six tubes. Observe carefully and compare the rate of hydrogen evolution in the several tubes. The reaction by which zinc liberates hydrogen may be expressed as



and consists in the transfer of two electrons from each atom of zinc to two hydrogen ions. Since the concentration of zinc is constant, the rate of reaction is naturally governed by the concentration of hydrogen ions. What can you conclude from your results as to the relative concentrations of hydrogen ions in the various mixtures? Are there any hydrogen ions in pure water?

Heat the first tube, containing zinc and concentrated sulfuric acid, and note that a gas is evolved (odor?) which is not hydrogen. It is due to the reaction of zinc with *molecules* of sulfuric acid. Equation.

(b) **Effect of the Solvent.** — Recall or test the action of a water solution of hydrogen chloride upon zinc and upon calcium carbonate (marble). Equations.

Obtain or prepare 10 cc. of a *dry* solution of hydrogen chloride

(d) **Test for Bromide.** — From parts (a) and (b) of this exercise deduce several characteristic tests for a bromide. Add silver nitrate solution to the solution of a bromide and test the solubility of the precipitate in ammonium hydroxide (?). Compare this with the chloride test — Exercise 27 (b).

EXERCISE 37

IODINE

Textbook: 254-260

yellow
(a) **Preparation.** — Repeat the first paragraph of Exercise 36 (a) using potassium iodide solution in place of the bromide (?).

Grind together 1 g. of potassium iodide and 2 g. of manganese dioxide. Transfer the mixture to a porcelain dish and moisten it with a solution of 1 cc. concentrated sulfuric acid in 2 cc. of water. Invert a clean dry funnel over this dish and gently heat it on a wire gauze (Fig. 18). Moist pieces of filter paper may be placed on the outside of the funnel to cool the surface upon which the iodine will condense. Note the color of the vapor and of the crystals. Write the equation for the reaction. When

a considerable amount of iodine has been collected scrape it into a clean watch glass and use it for the experiments below.

(b) **Physical Properties.** — Heat a crystal of iodine in a test tube. Does it melt? Sublime? Define sublimation.

Place a few iodine crystals in each of three test tubes. Add 10 cc. of water to the first and shake well, 1 cc. of alcohol to the second, and 1 cc. of solution of potassium iodide to the third (?). Is iodine as soluble in water as are chlorine and bromine? What is a solution of

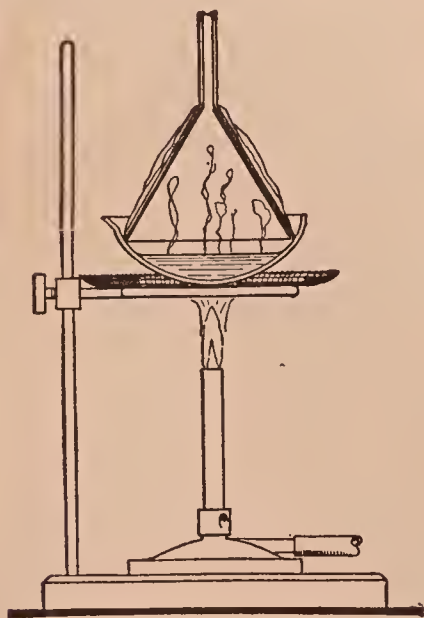


FIG. 18

iodine in alcohol called? *aqueous solution*

(c) **Starch Test.** — Prepare a starch suspension by boiling a pinch of starch in 10 cc. of water. Test the water solution of iodine with some of this (?). To some starch suspension add 1 cc. of a solution of potassium iodide (?); now add chlorine

water (?). Is starch a test for iodine in a compound? Dilute some of the starch suspension, add a drop of the alcoholic solution of iodine and dilute further until a transparent blue is obtained. Heat the contents of the tube to boiling (?); the color should reappear on cooling. How may this test be used conversely to test for starch in foods, baking powder, face powder, etc.?

(d) **Hydriodic Acid.** — To a few milligrams of potassium iodide in a dry test tube add several drops of concentrated sulfuric acid. Warm gently if necessary and note the color and odor of the gaseous products. Equations. What property of hydrogen iodide is shown here? Compare Exercise 36 (b). How may pure HI be prepared?

(e) **Test for an Iodide.** — From the foregoing experiments state how you would test for an iodide. To solutions of a chloride, a bromide, and an iodide add a solution of mercuric nitrate and from your results state how you could distinguish the three halides.

EXERCISE 38

HYDROGEN FLUORIDE (HYDROFLUORIC ACID)

Textbook: 260-262

Cover one side of a glass plate with a thin layer of paraffin by warming the plate uniformly at some distance above a flame and rubbing it with a piece of solid paraffin. When the paraffin has cooled, draw a figure or some letters with a pointed instrument in the center of the waxed surface, exposing the glass.

Place 5 g. of calcium fluoride (fluorspar) in a lead dish (store-room), moisten this with 2 or 3 cc. of concentrated sulfuric acid, stirring the mixture with a match stick, and cover the dish with the glass plate — waxed surface downward. (*Caution:* Do not breathe the fumes as they are very poisonous.)

Set this preparation away in the hood until the following day or later, then remove the paraffin and examine the plate for the etched design. The paraffin may be removed by warming the plate and wiping it with a towel or paper. Construct the equations for the preparation of hydrogen fluoride and for the etching of glass. Glass is a mixture of silicates; CaSiO_3 may be taken as representative.

Of what materials must containers for hydrofluoric acid be

made? Can hydrofluoric acid be oxidized to fluorine and water? What is the action of fluorine on water? Equation.

EXERCISE 39

PREPARATION OF PURE SODIUM CHLORIDE FROM ROCK SALT

Grind about 35 g. of rock salt in a mortar with 100 cc. of water. Transfer the whole to a beaker and warm with stirring to complete the solution of the salt. This will make an approximately saturated solution.

Dissolve 2 g. of sodium carbonate in 2 or 3 cc. of water and add this to the hot salt solution to precipitate the calcium and magnesium (?). Equation. Stir, allow the precipitate to settle, add a few drops of sodium carbonate solution and notice whether or not more precipitate forms. If it does, add more sodium carbonate solution to remove completely the calcium and magnesium.

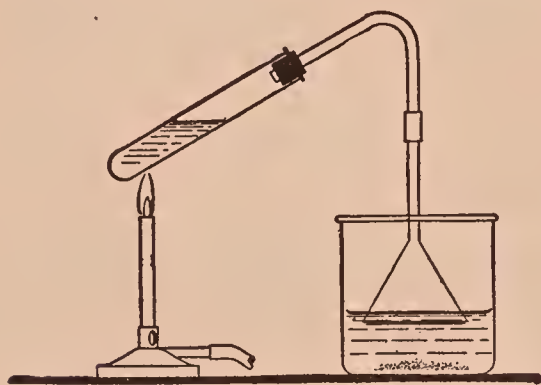


FIG. 19

Filter the hot solution, allow the filtrate to cool, and pass in HCl gas until a considerable amount of salt has precipitated. The HCl gas is conveniently supplied by heating some concentrated hydrochloric acid in a test tube provided with a one-hole stopper and delivery tube, bent to pass downwards. An inverted funnel should be attached to the delivery tube and so adjusted that the mouth of the funnel dips just below the surface of the salt solution (Fig. 19).

The effect of HCl in precipitating the salt from a saturated solution may be explained by the following equilibrium:

$\text{NaCl (solid)} \rightleftharpoons \text{NaCl (in solution)} \rightleftharpoons \text{Na}^+ + \text{Cl}^-$. Passing HCl gas into the solution is equivalent to adding Cl^- ions, hence the equilibrium is driven toward the left (see Textbook: 236–238).

When a sufficient amount of salt has precipitated, allow it to settle and decant the clear liquid. Using successive 10 cc. portions of dilute hydrochloric acid, wash the salt by decantation until the washings no longer give the barium chloride test for sulfates (?).

Transfer the solid to a filter and allow it to drain for some

time. Dry the salt by heating it carefully in a clean porcelain dish, stirring it with a glass rod. The salt should be perfectly white, finely crystalline, and should have a pleasant taste. Since the chlorides of calcium and magnesium have been removed, it is not hygroscopic and will remain dry in a damp atmosphere. Submit the product to your instructor for approval.

EXERCISE 40

ATOMIC STRUCTURE

Textbook: 279–299

Write clear definitions for the terms *electron*, *proton*, *atomic number*, *isotopes*. Contrast the proton and the electron with respect to *charge*, *size* and *mass* (weight). How many protons and how many electrons are there in a hydrogen atom? in a hydrogen molecule? in an oxygen atom? in a molecule of water? What are the atomic or molecular weights of these units of matter? Is the mass of each atom or molecule due principally to the protons or to the electrons? Does it appear that the *total* number of *protons* in an atom or molecule is numerically the same as the atomic or molecular weight? If this is true, what can you say as to the *total* number of *electrons* in an atom or molecule of weight *n*?

Draw a diagram consisting of concentric circles to represent the structure of each of the following atoms:

	<i>Atomic Number</i>	<i>Atomic Weight</i>
(a) Oxygen	8	16
(b) Sulfur	16	32
(c) Calcium	20	40
(d) Selenium	34	76
(e) Selenium	34	80
(f) Krypton	36	84

Let the inmost circle represent the *nucleus* of the atom. Indicate the number of protons and electrons contained in this, using appropriate symbols for proton and electron. Let each succeeding circle represent a *shell* of electrons and show the number of electrons which it contains. Show clearly the relation between atomic number and the composition of the nucleus, and between atomic number and the number of electrons outside the nucleus.

Define positive and negative valence in terms of electrons. From your atomic diagrams derive the positive or negative valence of each element you have pictured. Upon this basis predict the formula of the compound which selenium will form with calcium and the formula of a compound of selenium with oxygen. Explain the mechanism by which each combination will take place.

Describe the similarity which is apparent in your structures for oxygen, sulfur and selenium. What is the significance of this with regard to the relative positions of these elements in the periodic system?

Is the difference between two isotopes of an element due to a difference in the nuclei or to a difference in the electron shells? How do you account for the fact that the atomic weight of chlorine is approximately 35.5? Zinc has four isotopes having masses of 64, 66, 68 and 70. How many different zinc chlorides having the formula ZnCl_2 can be formed and what will be the molecular weights of the different species?

EXERCISE 41

THE PER CENT OF OXYGEN IN THE AIR

Textbook: 307-308

Determine the capacity of the graduated cylinder by filling it level full with water and withdrawing the water by means of a pipette to the 100-cc. mark. The last portion withdrawn can be measured in the 10-cc. graduate.

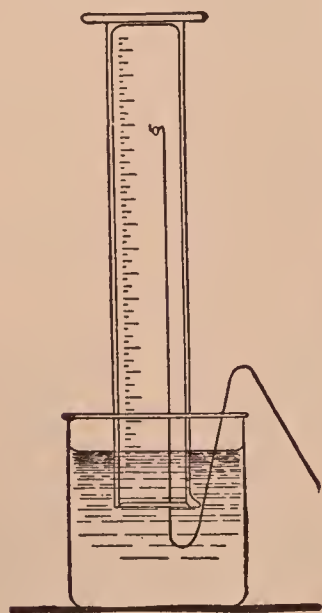


FIG. 20

Obtain a piece of white phosphorus in a dish of water, push the end of a piece of copper or iron wire through the phosphorus, place this upright over a beaker of water, and immediately cover it with the graduated cylinder. Figure 20 shows the arrangement of the apparatus. Be sure the open end of the graduate extends considerably below the surface of the water in the beaker and clamp the cylinder in this position. Using a form such as that suggested below, make a record in your notebook of the beginning conditions of the experiment and then set the

apparatus away in your desk until the next laboratory class. At that time adjust the levels of water inside the cylinder and in the beaker and take the readings necessary to complete your record.

Compare your result with the accepted value for the per cent by volume of oxygen in the air and calculate your percentage error. Write the equations to show what has happened to the oxygen in the cylinder.

TABLE OF DATA

Volume of air	cc.
Temperature	°C.
Barometric pressure	mm.
Vapor pressure of water	mm.
Corrected volume of air (S. C.) ...	cc.
Volume of gases left	cc.
Temperature	°C.
Barometric pressure	mm.
Vapor pressure	mm.
Corrected volume (S. C.)	cc.
Volume of oxygen removed (S. C.)	cc.
Per cent oxygen in air	%

EXERCISE 42

AMMONIA — PREPARATION; PROPERTIES

Textbook: 326-337

f. spoon

(a) **Preparation.** — In separate test tubes place 0.25 g. of (1) ammonium chloride, (2) ammonium nitrate, and (3) ammonium sulfate. To each tube add 2 cc. of sodium hydroxide solution. Warm each mixture, notice the odor and test the issuing gas with moist litmus paper and with a piece of filter paper moistened with concentrated hydrochloric acid (?). Grind a little quicklime or slaked lime with a small amount of an ammonium salt and test for ammonia as above (?). Equations. What general statement can you make concerning the preparation of ammonia? How would you test for an ammonium salt?

(b) **Solubility; Aqua Ammonia.** — Test for the presence of ammonia gas above a solution of ammonium hydroxide (?). What is your conclusion as to the stability of this compound?

white fumes
blue + pink
rapidly
ammonia

Fit a small flask with a stopper bearing a tube extending through in both directions as in Figure 21. Pour 2 cc. of concentrated aqua ammonia into the flask and warm this gently for a moment. Have a drop of water in that end of the tube which will extend into the flask, quickly insert the tube and stopper and invert the flask above a beaker of water to which a few drops of phenolphthalein have been added. Explain all that occurs.

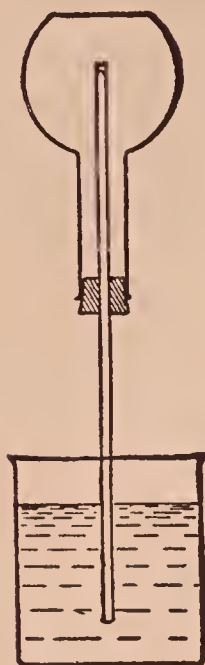


FIG. 21

(c) **Combination with Acids.** — In one test tube place 0.25 g. of sodium chloride and add a few drops of concentrated sulfuric acid. In another test tube place 0.25 g. of ammonium chloride mixed with an equal weight of powdered lime; heat this mixture and bring the two test tubes mouth-to-mouth (?). Write the equations.

Write equations to show the combination of ammonia with five acids and name the salt formed in each case.

(d) **Dissociation of Ammonium Salts; Diffusion.** — Place 0.5 g. ammonium chloride in a dry test tube and clamp it in an inclined position. In the mouth of the tube place moist pieces of red and blue litmus. Gently heat the ammonium chloride, note its apparent sublimation and observe the color changes of the litmus. Compare the relative weights of the two gases formed from ammonium chloride and explain the order of the litmus changes.

EXERCISE 43

NITRIC ACID — PREPARATION AND PROPERTIES

Textbook: 341-350

(a) **Preparation — Oxidizing Action.** — Place a gram of potassium nitrate in a test tube, add 1 cc. of concentrated sulfuric acid and insert a small plug of woolen yarn in the mouth of the tube. Support the test tube in a holder or clamp and warm the mixture until the vapor of nitric acid comes in contact with the wool (?). *wool caught on fire & burning brown fumes given off 4/10/23*

What other acids may be prepared by the action of sulfuric acid on their salts? Why may nitric acid be prepared by this method?

(b) **Action on Metals.** — Place in separate test tubes a small piece of the following metals: ^{2nd yr} zinc, ^{4th} copper, ^{5th} tin, ^{3rd} iron (nail), magnesium and aluminum. Add to each enough dilute nitric acid to cover the metal and note the relative speed with which each metal is acted upon — or the order in which the tubes begin to show the evolution of a gas. Add a few drops of concentrated nitric acid to any tube that fails to show any action.

According to your results arrange the metals in a column showing the relative ease with which they are oxidized by nitric acid. For comparison arrange these same metals in another parallel column according to their positions in the Electromotive Series (back of book or chart in classrooms). Note any difference of position in the two arrangements (?). **Note:** Nitric acid acts upon aluminum to form a continuous coating of insoluble Al_2O_3 which prevents any further action. Does this suggest a method of removing stains from aluminum utensils without harm to the metal?

In general, what are the products of the action of nitric acid on metals? Contrast the acid in this respect with sulfuric (dilute and concentrated) and with hydrochloric acid.

(c) **Decomposition.** — Heat 2 drops of concentrated nitric acid in a dry test tube and note the decomposition products. Equation. In the same manner heat a few crystals of copper nitrate or lead nitrate. Indicate how this method might be used to prepare the pure oxides of these metals. For what nitrogen compound does this suggest a method of preparation?

(d) **Test.** — An interesting test for nitric acid or nitrates, referred to as the “brown ring test,” may be carried out as follows: Dissolve one or two crystals of potassium nitrate in 3 cc. of water, add an equal volume of ferrous sulfate solution and mix well. Slightly incline the test tube and cautiously pour concentrated sulfuric acid down the inside of the tube. The acid sinks to the bottom and a colored layer forms where the two liquids meet.

Outline an alternative test for a nitrate, using concentrated sulfuric acid and copper.

(e) **Summary of Properties.** — Tabulate the properties of nitric acid as you did those of hydrochloric acid in Exercise 27. What is the essential difference in the chemical properties of these two acids?

EXERCISE 44

REDUCTION PRODUCTS OF NITRIC ACID

Textbook: 339-340; 346-348

43
wired up
as fte
F
(a) **Nitric Oxide.** — Place a gram of copper turnings in a test tube fitted with a stopper and bent delivery tube and arrange to collect several test tubes of gas by displacement of water. Add 3 cc. of dilute nitric acid (sp. gr. 1.2), clamp the tube in position and, if necessary, warm it to start the action. Explain why the gas in the generating tube is brown at first but becomes colorless as it passes into the collecting tube.

Thrust a glowing splinter into a test tube full of this gas (?). Compare the per cent of oxygen in this gas with the per cent of oxygen in air and explain the result just obtained.

Place another test tube full of nitric oxide mouth-to-mouth with a test tube full of air (?). Can you note any indications of temperature or volume changes in the reaction that occurs? Add 3 or 4 cc. of water to one of the tubes, place your thumb over the mouth of the tube and shake it until the gas becomes colorless. Remove the thumb for a moment and repeat the procedure (?). Test the solution with litmus paper and write equations to explain the changes observed.

Calculate the volume of nitric oxide which may be obtained by the action of nitric acid on a gram of copper.

(b) **Nitrogen Dioxide.** — Put a few drops of concentrated nitric acid in a test tube and drop into this a small piece of copper (?). Compare this with the preparation of nitric oxide (?). What other method of preparing nitrogen dioxide is suggested by reactions in (a)? Indicate how this gas could also be prepared from the nitrate of a heavy metal.

(c) **Ammonia.** — Place a few very small pieces of zinc in a 100-cc. beaker, add 2 cc. of water and 2 cc. of dilute nitric acid. Allow the action to continue for 5 minutes, then make the solution alkaline with sodium hydroxide solution and cover the beaker with a watch glass having a piece of wet red litmus paper adhering to its lower surface. Warm the mixture slightly if necessary, note the effect on the litmus and the odor of the gas in the beaker (?). Why did the ammonia not escape as it was formed? Write the equations.

Upon what two factors does the nature of the reduction product of nitric acid depend? Indicate six possible reduction products of nitric acid, each containing nitrogen with a different valence. Write separate equations to show the reduction of nitric acid by means of hydrogen to each of these products.

EXERCISE 45

NITROUS OXIDE

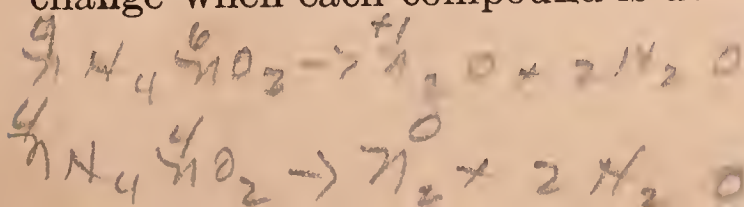
Textbook: 338

Heat 2 g. of ammonium nitrate in a clean test tube provided with a delivery tube as in Exercise 44 (a). The water over which the gas is collected should preferably be warm. (Why?) The ammonium nitrate should be heated gently until it melts and then caused to decompose slowly by holding the Bunsen burner in the hand and waving the flame under the test tube to produce a steady evolution of gas. If this substance is heated too rapidly it may explode.

Collect several test tubes full of the gas by displacement of water. The first should be discarded as it will contain air. Using a test tube full of the gas, devise a method of testing its solubility in cold water (?). Introduce a faint spark on the end of a wood splinter into a test tube full of the gas (?). Now introduce a brightly **glowing** spark into another tube full of the gas (?). How does nitrous oxide compare with oxygen as a supporter of combustion? What products would be formed if phosphorus were burned in this gas? What is the common name of nitrous oxide? *laughing gas*

Compare the percentage of oxygen in the three oxides of nitrogen with which you have experimented. Does the one containing most oxygen support combustion best? Explain. Calculate the weight of oxygen in a liter of nitrous oxide and in a liter of nitric oxide.

Contrast the decomposition products of ammonium nitrate with the products obtained by heating ammonium nitrite. Indicate the valence of each nitrogen atom in ammonium nitrate and in ammonium nitrite and show how these valences change when each compound is decomposed.



EXERCISE 46

NITRITES — NITROUS ANHYDRIDE

Textbook: 340–341

Heat 0.5 g. of sodium nitrate or potassium nitrate in a test tube until it decomposes, testing the decomposition product with a glowing splinter (?). Compare this result with the decomposition of two other nitrates — Exercises 43 (c) and 45. After the residue in the test tube has cooled add 1 cc. of dilute sulfuric acid (?). State how this would distinguish a nitrite from a nitrate — both will respond to the “brown ring test.”

— Melt a gram of sodium nitrate in a crucible, add a gram of “test” lead and continue heating the mixture for some time, stirring it with a rod or file. What compound of the metal is formed? What happens to the nitrate? When the crucible is cool add a small amount of water and dissolve the salt by warming and stirring. Pour this solution into a test tube, cool it and add dilute sulfuric acid. Note the color of the solution and of the gases evolved when the solution is warmed.

What is the action of nitrous anhydride on cold water? What happens when this solution is warmed? Equations.

EXERCISE 47

SULFUR — ALLOTROPIC FORMS

Textbook: 352–358

(a) **Changes on Heating.** — Fill a test tube one-fourth full of sulfur and heat it slowly until it boils, carefully noting all the intermediate changes. Allow the tube to cool, noting the reversal of the changes and save it for (c).

(b) **Rhombic.** — Shake 0.5 g. of sulfur in a test tube with 3 or 4 cc. of carbon disulfide and filter the solution into a clean watch glass. (*Caution:* Avoid flames.) Allow the solution to evaporate; watch the formation of crystals, noting their color and shape, and indicate the shape of a good crystal in your notes by means of a drawing.

(c) **Monoclinic.** — Slowly reheat the tube of sulfur from (a)

until the mobile stage of molten sulfur is just reached. Pour a few drops of this into a beaker of water and a small amount on a cold plate or dish. Compare the crystals formed with those prepared in (b).

(d) **Amorphous (Plastic).** — Continue heating the molten sulfur until the dark viscous stage is reached or even to the boiling point and then pour it into a beaker of cold water (?). Compare this product with the drops chilled in (c). Is the plastic form of sulfur stable? Leave it in your desk for a few days and examine it again (?). Test its solubility now and later in carbon disulfide (?). Above what temperature must molten sulfur be heated in order to form plastic sulfur when chilled? Why?

EXERCISE 48

SULFUR — COMBINATION WITH METALS; HYDROGEN SULFIDE

Textbook: 357-359

(a) **With Iron and with Copper.** — Prepare two small closed tubes about 12 cm. long as in Exercise 6 (b). Place a small amount of sulfur in the bottom of each tube and about 2 cm. above the sulfur place a wad of copper thread or fine wire in one tube and some iron picture wire in the other. In each case heat the tube about the metal until it is red-hot and then boil the sulfur. Notice any indications of chemical action as the sulfur vapor comes in contact with the hot metal (?).

Allow the tubes to cool, remove and examine the products and treat each sulfide with dilute sulfuric acid. If a gas is evolved notice its odor (?). Equations.

(b) **With Silver — Removal of Tarnish.** — Clean a small silver coin by washing it in hot sodium hydroxide solution followed by rinsing. Place a few drops of ammonium polysulfide, $(\text{NH}_4)_2\text{S}_x$, on the coin and note the formation of silver sulfide. The tarnishing of silver in the household is principally due to the formation of this substance from sulfur compounds in eggs, perspiration, and illuminating or cooking gas. The tarnish may be removed without harm to the silver by placing it in a hot salt solution in contact with a piece of aluminum — *e.g.*, an aluminum pan.

Pour 10 cc. of water into a beaker and add enough sodium chloride to make a saturated solution. Place the tarnished coin in this solution in contact with a piece of aluminum and boil the solution. If the method does not work the aluminum must be removed and cleaned by abrasion or by boiling it in a solution of sodium hydroxide for a moment. (Cf. Textbook: 585.)

EXERCISE 49

HYDROGEN SULFIDE — PROPERTIES AND USES

Textbook: 358–362

(a) **Preparation.** — Place a small piece of ferrous sulfide in a test tube and add 1 cc. of dilute hydrochloric or sulfuric acid (?). Cautiously note the odor of the gas and apply a flame to the mouth of the tube (?). Write equations to show the production and combustion of the gas.

(b) **Precipitation of Sulfides.** — Put 1 cc. of the solutions of metal salts listed below into separate test tubes and add 5 cc. of hydrogen sulfide solution to each (?). If no precipitate forms in certain tubes add ammonium hydroxide to those tubes (?). Finally test the solubility of each precipitate in dilute hydrochloric acid and tabulate your results as indicated below:

SOLUTION OF:	PRECIPITATE WITH H ₂ S? COLOR?	PPT. WITH H ₂ S + NH ₄ OH? COLOR?	SOLUBLE IN HCl?
Pb(NO ₃) ₂	yes brown		no
Mn(NO ₃) ₂			✓
CuSO ₄	yes black		no
ZnSO ₄	no milky	no white	yes
Na ₂ SO ₄	no soluble	" "	yes
FeSO ₄	no	yes dark green	no
SbCl ₃	yes brown		no
CaCl ₂			
AsCl ₃			

CLASSIFICATION

Three Groups of Sulfides

1. Precipitated in acid solution:
(Insoluble in dilute acid.) _____
2. Precipitated in alkaline solution:
(Soluble in dilute acid, insoluble in water.) _____
3. Not precipitated in either acid or alkaline solution:
(Soluble in water or decomposed by water.) _____

Write equations for any chemical changes which occurred.

(c) **Separation of Two Metals as Sulfides.** — To 5 cc. of water add 3 or 4 drops of an antimony chloride solution and just enough hydrochloric acid to dissolve the white precipitate (?). Now add a few drops of a solution of ferrous sulfate or ferrous chloride and 5 cc. of hydrogen sulfide water. Shake the mixture well and filter it. Make the filtrate alkaline with ammonium hydroxide and filter through another filter paper. What remains on each filter paper? Write the equations. As classified in (b), into what groups do these sulfides fall?

(d) **As a Reducing Agent.** — Put 1 cc. of a ferric chloride solution in a test tube, add some hydrogen sulfide water and warm gently. Note the change in the color of the solution and the formation of a fine precipitate. Write the equation.

Burn some sulfur in a bottle of air, add 10 cc. of water, shake and pour the solution (?) into a test tube. Add hydrogen sulfide water to this and explain the result. What changes in valence occur in this reaction?

To 1 cc. of a solution of potassium dichromate add a small quantity of hydrochloric acid and then hydrogen sulfide water until the solution changes color (?) and a milky precipitate results. Chromium is reduced from the dichromate to CrCl_3 . Indicate the valence changes and balance the equation.

EXERCISE 50

SULFUR DIOXIDE — PREPARATION; PROPERTIES

Textbook: 366-369

(a) **Preparation.** — Recall one method already employed in making sulfur dioxide (?). Obtain 0.5 g. sodium sulfite or

sodium bisulfite (formula?) in a test tube and add a few drops of dilute sulfuric acid. Test the evolved gas with moist blue litmus and note its odor by cautiously wafting some of it toward your nose.

Place 3 g. of copper turnings and 5 cc. of concentrated sulfuric acid in a test tube fitted with a one-hole stopper and a delivery tube bent to pass downward when the test tube is clamped in an inclined position. Gently warm the acid until it reacts upon the copper and collect two test tubes full of the gas by conducting it into the test tube, held in an upright position, until a litmus test or the odor of the gas at the mouth of the tube indicates that it is full. Stopper the tubes and save them and the generating tube for the following experiments.

Indicate by means of equations three different methods for preparing sulfur dioxide.

(b) **As a Bleaching Agent.** — Introduce some flower petals, pieces of green leaf or grass into one test tube of sulfur dioxide and again stopper the tube. After a time note any change that has occurred. Compare the bleaching action of sulfur dioxide with that of chlorine in regard to the type of chemical action, permanency and power.

(c) **As an Acid Anhydride.** — Add 3 or 4 cc. of water to the other tube of sulfur dioxide, quickly cover the mouth of the tube with the thumb and shake the tube. Note and explain the suction felt when the thumb is now removed (?). Test the solution with litmus paper. What has been formed? Note the odor of the solution. What is your conclusion with regard to the stability of the product? What other anhydride does sulfur form? Write equations to show the action of water on the two anhydrides.

Pour 5 cc. of a solution of sodium carbonate into a test tube and pass sulfur dioxide into the solution from the generator prepared in (a). After a short time the solution begins to effervesce. What is the gas? Equation. Continue passing in sulfur dioxide until effervescence ceases and evaporate the resulting solution to dryness in a porcelain dish. What remains? Treat this residue with a few drops of dilute sulfuric acid (?).

(d) **As a Reducing Agent.** — Saturate 10 cc. of water in a test tube with sulfur dioxide by passing in the gas at such a rate that the bubbles can be counted. Note that the bubbles of gas decrease in size and often disappear before they reach the

top of the solution. Explain. Continue passing in sulfur dioxide until bubbles of the gas rise through the solution with little or no decrease in size (?).

Divide this solution among three test tubes. To one portion add 2 cc. of bromine water (?) and to a second portion add, drop by drop, a solution of potassium permanganate until a delicate pink color persists in the solution. Now add a few drops of a solution of barium chloride and some dilute hydrochloric acid to each of the three tubes (?). For what ion is the barium ion a test? Write equations to show all that you observed.

EXERCISE 51

SULFURIC ACID

Textbook: 370-377

(a) **Preparation.** — Burn some sulfur, held in a deflagrating spoon, in each of two wide-mouthed bottles for a minute or two and cover the bottles.

Pour 10 cc. of water into one of the bottles, shake it and test the solution with litmus (?). Add a few drops of a solution of barium chloride and some dilute hydrochloric acid. To the second bottle of sulfur dioxide add a few drops of concentrated nitric acid. Place the bottle mouth-to-mouth with a bottle of air and pour the fumes back and forth (?). Then add 10 cc. of water, shake and add barium chloride solution and dilute hydrochloric acid as before.

Compare the results in the two bottles and indicate by means of an equation the action of the nitric acid. In what respects does this experiment resemble the lead-chamber process for the manufacture of sulfuric acid?

(b) **Temperature and Volume Changes on Dilution.** — Carefully measure 40 cc. of water in the graduated cylinder and pour it into a small flask. Measure exactly 10 cc. of concentrated sulfuric acid in the small graduated cylinder and pour the acid into the water in the flask. Note the heat produced and explain why it is safer to pour concentrated sulfuric acid into water rather than water into the acid. Cool the solution, measure it in the cylinder and calculate the per cent change in volume.

(c) **As a Dehydrating Agent.** — Try the effect of a few drops of concentrated sulfuric acid upon a pinch of sugar, bits of wood, paper and cloth. The formula of cane sugar is $C_{12}H_{22}O_{11}$; indicate the effect of concentrated sulfuric acid upon it. Take warning from these observations and avoid spattering sulfuric acid on clothing or skin. What would be the result if dilute sulfuric acid were spilled on cloth and allowed to dry?

(d) **As an Oxidizing Agent.** — Recall the action of hot concentrated sulfuric acid on copper (see Exercise 50). Test its action upon a small iron nail or piece of zinc (?). Compare these results with the action of *dilute* sulfuric acid upon the same metals. *Does not.*

(e) **Action on Salts.** — Recall the action of concentrated sulfuric acid on fluorides and chlorides (?). What is its action on a nitrate? What property of sulfuric acid makes it valuable for the preparation of other acids? Why can it not be used to prepare hydrobromic and hydriodic acids from bromides and iodides? Write the equation for its action upon KBr .

(f) **Solubility of Sulfates.** — Add a few drops of sulfuric acid to solutions of salts of lead, calcium, strontium and barium (?). Try the barium chloride test on a solution of a sulfate from the side shelf and also on a soluble carbonate and soluble phosphate, noting the effect first of the barium chloride, and then of the addition of dilute hydrochloric acid to the mixture in each of the three tubes.

EXERCISE 52

SODIUM THIOSULFATE

Textbook: 377-379

Prepare sulfur dioxide as in Exercise 50 and pass it into 5 cc. of a 10 per cent solution of sodium hydroxide until the solution is saturated. The solution should now give an acid test with litmus due to the presence of sodium bisulfite. Equation. Add more sodium hydroxide to this solution, a few drops at a time, with shaking, until the solution is just *slightly* alkaline. To this add 0.5 g. powdered sulfur, boil gently for at least 10 minutes and filter. Equations.

Test a small portion of the filtrate with dilute sulfuric acid; observe and explain a gradual change in the appearance of the

solution. Equation. Compare this with the action of an acid on a sulfite (?).

Pour about 2 cc. of water into a test tube, add to this 2 drops of silver nitrate solution and 2 drops of a solution of sodium chloride (?). Test the solubility of this precipitate in some of the thiosulfate solution. State the application of the observed result to photography.

Stain a piece of cloth with a drop of tincture of iodine or dip the cloth in chlorine water and note that the cloth retains the chlorine odor. Now dip the cloth in some sodium thiosulfate solution and note the effect (?). Suggest the practical application of the result.

EXERCISE 53

NORMAL SOLUTIONS — EQUIVALENT WEIGHTS

Textbook: 379-382

Write in your notebook answers to the following:

1. What is a standard solution? A molar solution? A normal solution? An equivalent weight? (Refer to Exercise 15.) Illustrate each of these definitions by means of an example.
2. How much is a gram equivalent of phosphoric acid? Of $\text{Al}_2(\text{SO}_4)_3$?
3. A gram molecule of phosphoric acid contains how many gram equivalents? How many liters of normal solution would it make? It would make 2 l. of what normality?
4. What volume of a base of 0.25 normality (N/4) will be neutralized by 100 c. of a twice normal (2N) acid?

Problem. — (a) If a liter of N potassium hydroxide solution is treated with hydrochloric acid and evaporated, what weight of salt remains? If 99.4 g. of this salt are obtained in this way from a liter of potassium hydroxide, what was the normality of the base?

(b) If 100 cc. of an unknown acid neutralize 150 cc. of the base of the above strength, what is the normality of the unknown acid?

EXERCISE 54

NORMAL SOLUTIONS — ACIDIMETRY

Textbook: 380-382

Quantitative — Formal Report Required

Measure 65 cc. of a 10 per cent solution of sodium hydroxide into a clean bottle, add enough water to dilute the solution to 150 cc. and mix well. This solution should now be slightly stronger than normal.

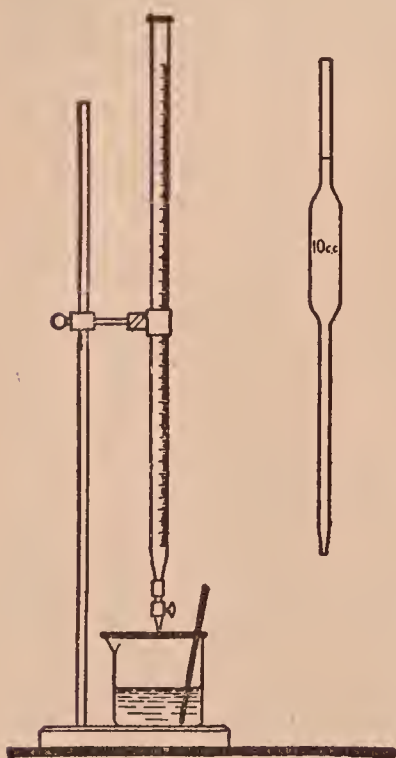


FIG. 22

Weigh accurately a clean, dry, 30-cc. porcelain dish. Measure exactly 10 cc. of the sodium hydroxide solution into the weighed dish by means of a pipette. To use the pipette, suck the liquid above the 10-cc. mark, being careful not to draw it into your mouth, quickly place index finger over the upper end of the pipette and by carefully releasing the finger allow the liquid to run down until the meniscus is **exactly** at the mark. Then allow this 10 cc. of solution to run from the pipette into the dish, touching the tip of the pipette to the inner surface

of the dish to remove the last drop.

Add 5 cc. of dilute hydrochloric acid to the solution in the dish, place the dish on a water bath and evaporate the contents to apparent dryness. Now cover the dish with a watch glass and heat it gently on a wire gauze until no more moisture condenses on the watch glass, then heat fairly hot. When the dish is quite cool weigh it again. From the weight of the salt obtained, calculate the weight of sodium hydroxide in 10 cc. of your solution and from this the normality of the solution. Is it necessary to know the exact amount of hydrochloric acid added provided an excess was used? Why?

With a Report Card, provided by your instructor, and a **clean, dry, 100-cc.** graduated cylinder obtain a sample of “unknown” acid. Set up a burette as in Figure 22, wash it with water, rinse with a small amount of your sodium hydroxide solution,

and fill the burette with this solution. Make sure that all the air bubbles have been displaced from the tip of the burette and its rubber connection. This may be accomplished by bending the tip upward and allowing a little of the solution to flow. Finally, adjust the bottom of the meniscus to the zero reading of the burette.

Wash and dry the 10-cc. pipette or rinse it with some of the unknown acid. Accurately measure 10 cc. of the unknown acid in the pipette, and transfer it to a 100-cc. flask and dilute it with water to about 50 cc. Add 2 or 3 drops of a solution of methyl orange or phenolphthalein. Recall the color changes of these indicators — Exercise 27 (*d*). Now carefully run in the sodium hydroxide solution from the burette until the end-point is exactly reached. The last drop of solution added should cause the indicator to change color. The solution must be stirred or shaken after the addition of each small portion of sodium hydroxide solution. Repeat this titration several times with 10-cc. portions of the unknown acid and average your results.

From the volume of the sodium hydroxide solution required and its normality, calculate the normality of the unknown acid — *i.e.*, how many cubic centimeters of an exactly normal acid are equivalent to one cubic centimeter of the unknown acid.

EXERCISE 55

PHOSPHORUS

Textbook: 384-397

(*a*) **Allotropy.** — From a piece of glass tubing prepare a tube about 8 cm. long and closed at one end. Place a few milligrams of red phosphorus in this tube and heat it until some of the phosphorus has distilled and condensed farther up the tube. When the tube is cool, cut it under water at this point and remove some of the distilled phosphorus by means of a wire or the forceps. (*Caution:* White phosphorus ignites in air and if in contact with flesh produces painful burns which heal very slowly. It should not be handled with the fingers.) Dry this on a filter paper and leave it exposed to the air until it ignites. Contrast the two forms of phosphorus with respect to several properties.

(b) **Three Types of Phosphates.** — Place about 0.2 g. of disodium phosphate, $\text{Na}_2\text{HPO}_4 \cdot 12\text{H}_2\text{O}$, in a porcelain crucible and heat it to redness for some time. Equation. Allow the product to cool and dissolve it in water. To this solution add a few drops of silver nitrate solution (?). *muddy*

Heat about 0.2 g. of microcosmic salt, $\text{NaNH}_4\text{HPO}_4 \cdot 4\text{H}_2\text{O}$, in a crucible as above. What is evolved? Equation. When cool dissolve the product and add silver nitrate solution (?). *muddy*

yellow
sal Test a solution of disodium phosphate with silver nitrate solution (?). Test the solubility of half of the precipitate in an excess of a dilute acid and the other half in ammonium hydroxide (?). Write equations for the reactions of silver nitrate with sodium pyrophosphate, sodium metaphosphate, and disodium orthophosphate. Compare the colors and forms of any precipitates so produced.

(c) **Phosphoric Acid.** — Slowly heat a minute quantity of red phosphorus with 1 cc. of concentrated nitric acid in a porcelain evaporating dish until brown fumes are no longer evolved. When cool take up the residue with 2 or 3 cc. of water, filter if necessary and test the solution with silver nitrate solution. According to the color of the precipitate (cf. b) which phosphoric acid have you obtained? Equation.

(d) **Calcium "Superphosphate."** — To 5 g. of bone ash (tertiary calcium phosphate) in a porcelain evaporating dish add 3 cc. of water and 3 cc. of concentrated sulfuric acid. Warm the mixture for 10 minutes stirring occasionally. Add 10 cc. of water, stir and filter the solution. Neutralize the solution with ammonia and then make it just **slightly** acid with dilute HNO_3 . Test the resulting solution with silver nitrate solution (?). Why was it necessary to neutralize the excess acid and also to avoid an ammoniacal solution — refer to (b)?

What is the correct chemical name for the "superphosphate"? For what is the substance used? Why is it preferable to bone ash for this purpose?

EXERCISE 56

ARSENIC AND ANTIMONY

Textbook: 399-412

(*Caution:* Arsenic and antimony compounds are very poisonous.)

(a) **Reduction of Arsenic Trioxide.** — Prepare a tube as in Exercise 55 (a). Place a few milligrams of arsenic trioxide in the bottom of the tube, cut a piece of charcoal to fit into the tube and place this about 1 cm. above the trioxide in the tube. Heat the charcoal red-hot and, while keeping it hot, heat the arsenic trioxide to cause its vapor to pass over the charcoal. What is the black ring formed above the charcoal? Heat the black deposit and note whether it melts or sublimes (?). Heat some powdered antimony in a small glass tube and compare its behavior with that of arsenic.

(b) **Arsenious Sulfide and Ammonium Sulfarsenite.** — Dilute 1 cc. of arsenic trichloride in a test tube with water and add some hydrogen sulfide solution (?). Allow the precipitate to settle, pour off most of the supernatant liquid and test the solubility of the precipitate in a mixture of ammonium hydroxide and hydrogen sulfide solution, or in a solution of ammonium polysulfide, $(\text{NH}_4)_2\text{S}_x$. If the latter is used, ammonium sulfarsenate is formed. Write the equations. Repeat the experiment using lead nitrate solution in place of arsenic trichloride and from your results indicate how the sulfides of lead and arsenic could be separated.

(c) **Colloidal Arsenious Sulfide.** — Boil a small quantity of arsenious oxide with 20 cc. of distilled water and filter the solution. To the filtrate add some hydrogen sulfide solution. What reaction do you expect? Is the product visible? Compare with the result in (b) when H_2S was added to a solution of AsCl_3 and explain the difference. To different portions of the colloidal solution just obtained add a few drops of the following solutions: sodium chloride, hydrochloric acid, acetic acid, sugar. Explain any difference in the results and classify as electrolytes and non-electrolytes the solutions which were added.

(d) **Displacement.** — Put 2 cc. of a solution of antimony trichloride in a test tube, add an equal volume of water and

enough hydrochloric acid to dissolve the precipitate (?). Drop a piece of tin into this solution and watch the action for a few minutes (?). Drop a piece of tin into a solution of arsenic trichloride and compare the deposit formed on the tin in the two cases.

Study this type of reaction with a piece of zinc in a solution of lead nitrate and a clean iron nail in a solution of copper sulfate. Write equations for all the above changes. What metals could be displaced from solution by copper? Make a general statement with regard to the displacement of a metal from solution by another metal.

(e) **Amphoteric Character.** — Add some ammonium hydroxide to 1 cc. of antimony trichloride solution. Divide the precipitate into two parts: to one add hydrochloric acid, to the other a solution of sodium hydroxide. Write the equations. Is Sb(OH)_3 an acid or a base? What kind of oxide is Sb_2O_3 ? What kind of an element is antimony?

(f) **Antimony Sulfide.** — To 1 cc. of a solution of antimony trichloride add some water and note the hydrolysis. Add a few drops of hydrochloric acid (?) and then add some hydrogen sulfide solution (?). This sulfide is similar to As_2S_3 in its action with ammonium sulfide (cf. b). Equation. Pour off the supernatant liquid and dissolve the precipitate by adding a few drops of concentrated hydrochloric acid. Show that this is a reversible reaction.

EXERCISE 57

BISMUTH

Textbook: 412-415

(a) **Basic Bismuth Nitrate.** — Warm 0.1 g. of metallic bismuth in about 1 cc. of concentrated nitric acid until solution occurs. Equation. To the resulting solution add water, drop by drop, until a precipitate forms (?). What is the pharmaceutical name of this product and for what is it used?

(b) **Reduction of the Basic Oxide.** — To 1 cc. of a solution of bismuth trichloride add some ammonium hydroxide and filter off the precipitate. Prepare some sodium stannite, Na_2SnO_2 , by adding sodium hydroxide to 1 cc. of stannous chloride until the precipitate formed at first is dissolved in the excess sodium hydroxide. Add this solution to the precipitate of BiO(OH) on

the filter paper (?). The reduction product is metallic bismuth. Equation.

P- ✓ (c) **Bismuth Sulfide.** — Pour 1 cc. of a solution of bismuth chloride into a test tube, add water and if a precipitate forms (?) dissolve it by adding dilute hydrochloric acid. Now add hydrogen sulfide solution and compare the precipitate with the corresponding compounds of arsenic and antimony.

Is bismuth more or less metallic than antimony? Give several reasons for your answer. How would you distinguish a solution of SbCl_3 from a solution of BiCl_3 ?

EXERCISE 58

SILICON

Textbook: 417-430

(a) **Reduction of Silica — Hydrogen Silicide.** — Mix a gram of fine sand with the calculated amount of magnesium powder required to reduce the silica to silicon and to combine with the latter to form magnesium silicide. Place the mixture in a test tube and heat it gradually until a reaction occurs (?) and then heat it as hot as possible for a few minutes. Be careful not to point the test tube at anyone while heating it. Allow the product to cool, break the tube and cautiously throw some of its contents into a small beaker containing 5 cc. of concentrated hydrochloric acid (?). Write the equations.

(b) **Soluble Glass — Silicic Acid.** — In a porcelain crucible mix one gram of infusorial earth ("kieselguhr," formula?) with 2 g. of sodium carbonate, 2 g. of potassium carbonate, and a piece of solid potassium hydroxide (about 2 g.). Heat this mixture as hot as you can for at least 15 minutes. When the crucible is cool, place it with its contents in a small beaker, add 25 cc. of water and boil for some time to dissolve the soluble portion. Filter this solution and divide the filtrate into two portions.

To one portion of the filtrate add 5 cc. of concentrated hydrochloric acid, drop by drop, stirring the solution (?). Equation. To the other portion add 5 cc. of concentrated hydrochloric acid all at once (?). Evaporate this solution almost to dryness in a dish or beaker (?). If the same product results here as in the other portion why did you not see it at first? What is the final product that may be obtained by heating this residue?

What is sodium silicate commonly called? Mention several of its uses.

(c) **Glass.** — Make a small loop at the end of a platinum wire which is fused into a piece of glass tubing at its other end. The glass tubing is to serve as a handle for the wire. Heat the loop for a moment in the Bunsen flame and dip it into some powdered sodium carbonate. Heat again and repeat the operation until the sodium carbonate forms a clear bead in the loop. Touch the bead while hot to some powdered silica and heat the bead as hot as possible in order to dissolve the silica. Likewise take up some powdered calcium carbonate on the bead and continue heating and dipping the bead in the different substances until a clear fusible bead is obtained. This bead should now have a composition similar to that of ordinary glass. Write the equations for the reactions which have occurred.

Grind a few fragments of glass to a powder in the mortar, moisten this powder with a *few drops* of water and add to it a drop of phenolphthalein solution (?). Why does not water in an ordinary glass vessel give this test?

Mention three advantages of fused quartz as compared with ordinary glass. How may glass or quartz be etched? Write equations to illustrate this. (Recall Exercise 38.)

EXERCISE 59

BORON

Textbook: 431-434

(a) **Boric Acid.** — Dissolve 5 g. of borax in 15 cc. of hot water in a small beaker. Add the calculated amount of dilute sulfuric acid to convert the borax to boric acid, assuming 1 cc. of dilute sulfuric acid to contain 0.25 g. H_2SO_4 . Allow the solution to cool and examine the product. For what is it used? What is the anhydride of boric acid? Of what acid is borax a salt? What is its anhydride? Is boric acid more or less hydrated than the acid corresponding to borax?

(b) **Borax Glass — Bead Tests.** — Make a loop about 3 mm. in diameter in the end of a platinum wire as in Exercise 58 (c). Heat the loop for a moment in the Bunsen flame and dip it into some powdered borax. Heat again until the borax which ad-

hered to the hot wire melts to a clear bead in the loop. Touch this bead while hot to some minute particles of cobalt oxide or cobalt nitrate and heat again until the particles dissolve in the bead. The resulting color is characteristic of cobalt glass. Other metallic oxides dissolve in borax glass to give characteristic colors. The borax bead test may be repeated with compounds of copper, iron, nickel and manganese. In some cases a different color is obtained for the same metal in the oxidizing and reducing zones of the flame.

What properties of a "boro-silicate" glass make it superior to ordinary glass for chemical apparatus and for cooking ware?

(c) **Tests for Borates.** — Place a small quantity of borax or boric acid in a porcelain dish, moisten this with some concentrated sulfuric acid, and add a few cubic centimeters of alcohol. Light the alcohol in the open dish and observe the color of the flame as contrasted with the ordinary flame of alcohol.

Saturate a few cubic centimeters of water with borax and add dilute hydrochloric acid to this solution until it is acid to litmus. Moisten a piece of turmeric paper with this solution, note its color and dry it on a watch glass over a beaker of boiling water. When the turmeric paper is dry touch it with a drop of sodium hydroxide solution, by means of a glass rod, and note the change in color.

EXERCISE 60

CARBON

Textbook: 436-454

(a) **Preparation of Charcoal.** — Place a small piece of wood in an iron crucible having a diameter of about 3 cm. Cover the crucible and heat it on a clay triangle for several minutes with the full flame of the Bunsen burner. Try to determine whether or not the escaping products are combustible. When the crucible is cold examine the residue (?).

(b) **Preparation of Coke.** — Repeat (a) using about 2 g. of coal instead of the piece of wood.

(c) **Adsorptive Power of Charcoal.** — Dilute a drop of methyl violet to 10 cc. ("indelible" pencils contain this dye), add 2 g. of animal charcoal (bone black), boil and filter the solution (?).

Suggest some practical application of this property of finely divided carbon.

(d) **Carbon Dioxide — Carbonates.** — Fit a test tube with a one-hole stopper bearing a delivery tube bent twice at right angles so as to pass downward. Place about 3 g. of marble in the test tube and add 5 cc. of dilute sulfuric acid. Insert the stopper with the delivery tube and allow the gas to bubble into 2 cc. of sodium hydroxide solution for a short time (?). Now add dilute sulfuric acid to this solution (?). Equations. Of what acid is carbon dioxide the anhydride?

Take 5 cc. of clear limewater in a test tube, add an equal volume of water and pass carbon dioxide into the solution until the turbidity which appears at first has been cleared up (?). Boil the solution (?). Explain the relation of this result to the formation of a deposit in teakettles and boilers when certain hard waters are used.

EXERCISE 61

IDENTIFICATION OF A SALT — A REVIEW

Purpose :

1. To discover the acid radical present in an unknown substance.
2. To test whether or not the substance is a hydrate.
3. To test whether or not the substance is an ammonium salt.

Procedure:

- (a) Devise and write equations for distinguishing reactions of salts of the following acids:

HCl, HBr, HI, HNO₃, HNO₂, H₂S, H₂SO₃, H₂SO₄, H₂S₂O₃, H₃PO₄, H₃BO₃, H₂CO₃.

For example, any chloride with concentrated sulfuric acid and manganese dioxide gives chlorine gas which may be recognized.

Next devise a test for an ammonium salt and also for a hydrate.

- (b) Present this outline to your instructor for approval. Try all the identifying tests with known substances from the shelf, recording in your notebook such phenomena as precipitates (color), gases evolved (color and odor), etc.

- (c) Your instructor will now give you a Report Card and you may obtain an "unknown" from the storeroom. Analyze this substance by the tests you have outlined.
- (d) Record your results in your notebook and on the Report Card and return the latter to the storeroom.

EXERCISE 62

A REVIEW OF OXIDATION-REDUCTION

Equations by the Ion-Electron Method

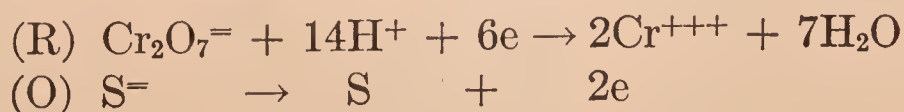
Textbook: 201-203

Define oxidation and reduction in terms of valence change. Recall the idea of positive and negative valence arrived at in Exercise 40 (cf. Textbook, Chapter 17) and define oxidation and reduction in terms of a loss or gain of electrons.

What is the essential difference between an atom of sodium and a sodium ion? Is oxidation or reduction involved when a metal ion, such as the cupric ion, is deposited in the electrolysis of a cupric chloride solution? Explain. What must happen to a sulfur atom in order that it may become a sulfide ion?

Refer to the third paragraph of Exercise 49 (d) and write the valence changes and the balanced equation for the action which occurred. Recalling that most acids, bases, and salts are practically completely ionized, it is apparent that in this reaction there are certain ions which are present both before and after the chemical change but which have taken no part in the reaction. To which two ions in this reaction does the above statement apply? The equation can, therefore, be formulated from the other ions and the electrons which are lost and gained by these ions, omitting entirely the unchanged ions. It is most convenient to do this in two *partial* equations, one involving the loss of electrons, the other the gain of electrons. If this loss and gain is equalized by multiplying the partial equations by the necessary factors and the equations are added, the electrons cancel out and the balanced ionic equation is obtained.

Illustration: The equation to which we have referred should be formulated by this method as follows:



Multiplying the oxidation (O) partial equation by 3 and adding the result is

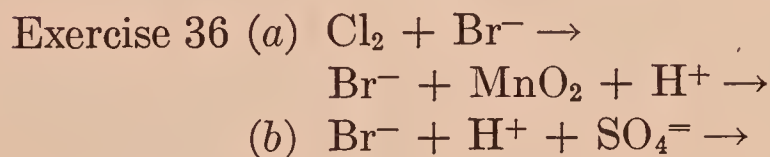
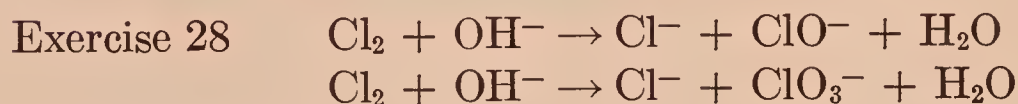
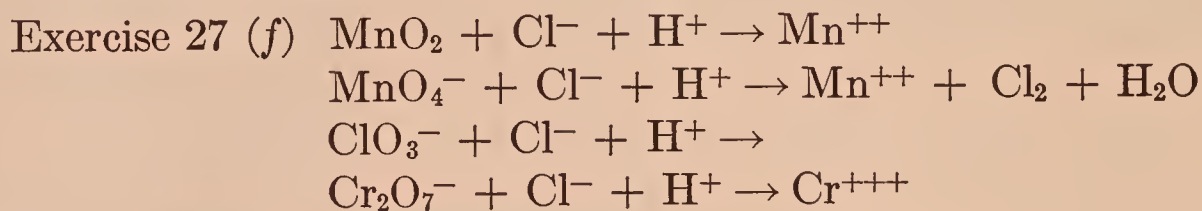


This balanced ionic equation has advantages over the usual molecular equation in that:

(1) Only those ions appear which are actually involved in the reaction.

(2) The equation is general — that is, it represents the reaction of *any* dichromate with *any* sulfide in the presence of *any* acid.

By means of the method just outlined write the ionic equations for the oxidation-reduction reactions which you observed in the following experiments:



Exercise 44 (a, b, c)

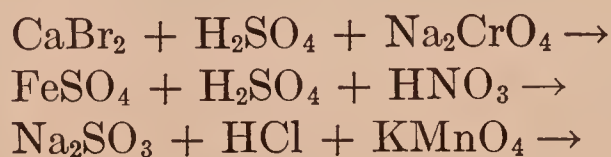
Exercise 50 (c)

Compare the final ionic equation in each case with the molecular equation which you wrote previously, noting the ions which were inactive.

Write the ionic equation for the action of nitric acid upon a sulfide to produce nitric oxide and free sulfur. Translate this into a molecular equation for copper sulfide.

Formulate the ionic equation for the action of a halogen (X) with a hot, concentrated base. Translate this into a molecular equation for iodine and barium hydroxide.

Write the ionic equations for the following reactions, stating which ions are inactive.



PART II

THE METALS

EXERCISE 63

THE PHYSICAL PROPERTIES OF THE METALS

Textbook: 522-531

Refer to the table of physical properties of the metals and to your textbook in answering the following questions.

1. Contrast the **physical properties** of the metals and non-metals in at least three particulars.
2. State the physical property of the metal concerned which makes possible the use indicated:

gold and silver thread for cloth; gold leaf for lettering;
aluminum in aeroplane frames; lead for plumbing;
lead for bullets and shot; copper for trolley wires.

3. Which metal has the highest melting and boiling points? What use is made of the metal because of this fact?
4. Which metal has a melting (or freezing) point below ordinary temperatures? What use of this metal depends upon this fact? How does the boiling point of this metal compare with the boiling points of other metals? Do you know of any appliance in which the vapor of this metal is used?
5. Will the following metals float or sink in mercury: gold, silver, platinum, lead, tungsten, iron?
6. Which is the heaviest metal listed in the table? How does its melting point compare with the melting points of other common metals? What use for the metal does this suggest? Mention other uses of this metal.
7. How many pounds should a "gold brick" weigh if it measures 20 cm. $\times$ 10 cm. $\times$ 10 cm. and is real? What would it weigh if it were composed of lead with a thin plating of gold? (1 lb. = 454 g.; 1 kg. = 2.2 lb.)
8. Mention two metals which can be melted in a tube placed in boiling water. Mention two metals which will float on water.

9. Which seven metals were known and used in very ancient times? Which of the metals commonly used in the household is of most recent discovery?
10. Does there appear to be any general relationship between the ability of a metal to conduct heat and its ability to conduct electricity? Illustrate your answer by means of at least two instances.
11. Which metal is the best conductor of heat and electricity? Which metal is most commonly employed for electrical conduction? Why do you give a different answer to these two questions?
12. The conductivity comparisons in the table are for wires of the same **size**; if the comparison is made on the basis of wires of the same **weight** for a given length, which metal is the best conductor? Suggest some reasons why this metal is not more commonly used for electrical conduction.
13. From the data in the table suggest two or more **physical** reasons why aluminum is a good material for cooking utensils. Assuming other conditions the same, will water boil more quickly in a vessel of iron, platinum, copper, or gold? Give three **physical** reasons why lead would be an undesirable material for a frying pan.

EXERCISE 64

PROPERTIES OF ALLOYS

Textbook: 531-534

(a) **General.** — Alloys are mixtures of two or more metals, solid solutions of metals in each other, or, in some cases, definite compounds of one metal with another. Alloys are usually prepared by melting together the component metals. The physical properties of an alloy may be intermediate to, or greater than, or less than the corresponding values of the component metals.

Referring to the table of physical properties, what do you conclude as to the general effect of alloying upon the following properties of the metals: density, melting point, conductivity? Particular care is taken to remove traces of other metals from copper which is to be used for electrical conduction. Can you suggest the reason for this?

PHYSICAL PROPERTIES OF THE METALS

METAL	DENSITY OR SP. GR. (H ₂ O = 1)	MELTING POINT °C.	BOILING POINT °C.	ELECTRI- CAL RE- SISTIVITY 20°C.	THERMAL CONDUCT- TIVITY	DATE OF DIS- COVERY
Aluminum	2.7	658.7	1800	2.82	.504	1828
Antimony	6.62	630	1440	41.7	.042	1450
Arsenic	5.73	Sublimes	450	33.3	—	1694
Barium	3.8	850	950	—	—	1808
Bismuth	9.78	269.2	1436	120.	.0194	1450
Cadmium	8.65	320.9	778	7.6	.222	1817
Calcium	1.54	810	—	4.6	—	1808
Chromium	6.92	1615	2200	2.6	—	1797
Cobalt	8.72	1480	—	9.7	—	1773
Copper	8.94	1083	2310	1.72	.918	Prehist.
Gold	19.32	1063	2500	2.44	.700	Prehist.
Iron	7.86	1530	2450	10.	.161	Prehist.
Lead	11.34	327	1525	22.	.083	Prehist.
Magnesium	1.74	651	1120	4.6	.376	1829
Manganese	7.42	1230	1900	5.	—	1774
Mercury	13.6	-38.85	357	95.78	.0197	Prehist.
Nickel	8.75	1452	—	7.8	.142	1751
Platinum	21.37	1755	3910	10.	.166	1741
Potassium	.87	62.3	712	7.2	—	1807
Silver	10.5	960.5	1955	1.59	.990	Prehist.
Sodium	.97	97.5	750	—	—	1807
Tin	7.29	232	2270	11.5	.155	Prehist.
Tungsten	18.7	3400	5830	5.6	.476	1781
Zinc	7.15	419.4	930	5.8	.265	1520
ALLOYS						
Brass						
70Cu:30Zn	8.6	900		7.	.260	
Bronze						
67Cu:33Sn	8.6	750				
Gold coin						
90Au:10Cu	17.17	940				
Solder						
67Pb:33Sn	9.4	240		16.		
Steel						
99Fe:1C	7.7	1510		12.	.115	

ORDER OF DUCTILITY OF THE COMMON METALS

Gold, silver, platinum, iron, copper, zinc, tin, lead.

Except for iron, the malleability of the metals is also in the above order.

(b) **Determination of the Melting Point of an Alloy.** — An alloy known as Wood's metal consists of Bi, 50%; Pb, 25%; Sn, 12.5%; Cd, 12.5%. Refer to the table and tabulate the melting points of these metals in your notebook. Obtain 40 to 50 g. of Wood's metal from the storeroom and determine its melting point as follows:

Place the alloy in an ordinary test tube and clamp this in an upright position in a beaker of boiling water. Place a 100° thermometer in the Wood's metal and stir it gently as it melts. When the alloy is completely molten and the thermometer registers about 90°, turn out the gas flame and remove the beaker of water. Let the thermometer remain stationary in the melted metal and read the temperature every 30 seconds as the alloy cools and freezes. Continue the readings for several minutes after the alloy has solidified. Record time and temperature in your notebook in parallel vertical columns as you take the readings. Your temperature readings should descend from about 85° to about 60° and will require 10 to 15 minutes.

Do not attempt to remove the thermometer from the solidified mass, but melt the latter in the beaker of boiling water as before. Remove the thermometer and carefully detach any metal which adheres to it. While the alloy is in the molten state pour it into a beaker of cold water. If any of the alloy remains in the test tube, allow it to cool and scrape it out by means of a piece of glass tubing. Return the Wood's metal to a bottle provided for that purpose.

Construct a form in your notebook as in Figure 23 (graph paper may be used) and from your data plot the "cooling-curve" of Wood's metal. Draw a line through the points obtained and indicate the melting (freezing) point of the alloy. How does your result compare with the average of the melting points of the component metals?

What is meant by the term, "Heat of Fusion"? Explain how this is the cause of the "halt" in the cooling-curve of a molten metal or alloy.

(c) **Amalgams.** — Alloys containing mercury* are called amalgams. All the common metals except iron form amalgams. Contact of the metal with mercury, or contact of a

* *Caution.* — All soluble compounds of mercury are extremely poisonous. Mercury vapor, if breathed, causes slow poisoning which is cumulative in effect. There is considerable belief that there is danger of slow poisoning from amalgam dental fillings.

more active metal with a solution of a mercury salt will produce an amalgam. Is there danger of injuring gold or silver jewelry when working with metallic mercury? When working with mercury salt solutions? If the jewelry contains a con-

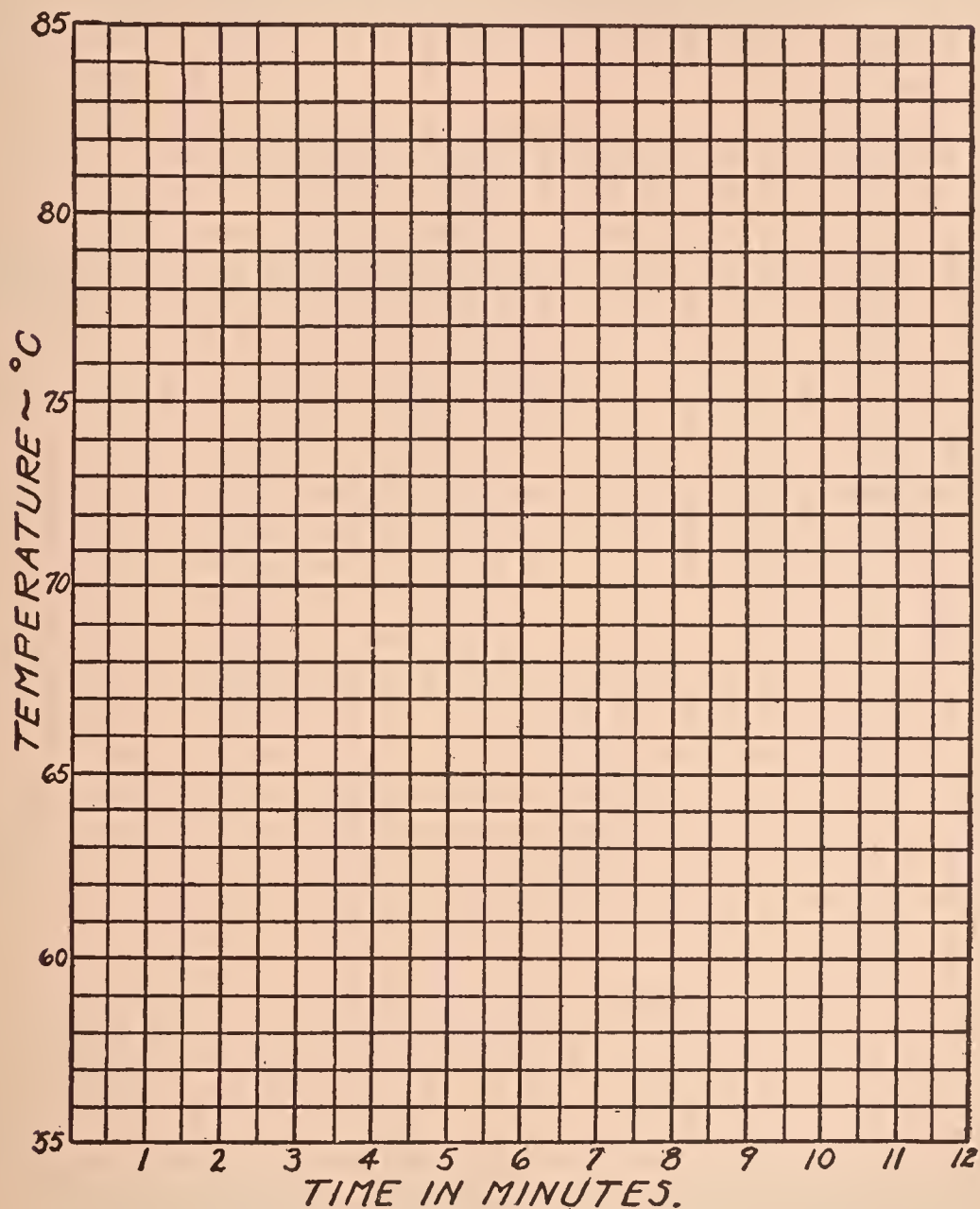


FIG. 23

siderable proportion of copper would your answer to the second question be different?

Dip a piece of copper or a copper coin into a solution of a mercuric salt until it has a gray coating. Remove the copper and polish it with a towel or piece of filter paper. Describe the result. The mercury will slowly diffuse into the copper. Devise a simple method for removing the mercury from the copper and, if your instructor approves, proceed.

EXERCISE 65

THE CHEMICAL PROPERTIES OF THE METALS

Textbook: 525-528

(a) **General Properties.** — Contrast metals and non-metals with respect to:

- (1) The action of water on their oxides.
- (2) The action of water on their chlorides.
- (3) The nature of the ions in which they are usually found and the pole at which they are liberated in electrolysis.

Illustrate (1) and (2) by means of equations and (3) by definite examples.

(b) **Displacement Series.** — By means of appropriate experiments determine the relative order of activity of the following metals:



For example, if metal A, placed in a solution containing ions of metal B, partly dissolves, displacing B, then metal A is more active than metal B and belongs above it in the series. Assuming both metals to be bivalent, the change which occurs may be expressed:



(c) **Amphoteric Metals.** — Certain elements, usually classified as metals, are sometimes found in the cation and sometimes in the anion portion of their compounds. Such elements are said to be amphoteric, that is, capable of forming both acids and bases. Common examples of such metals are: Al, As, Cr, Mn, Sb, Sn, Zn.

Obtain solutions of salts of Al, Sb, Sn, and Zn in which these metals form the cations. Use about 3 cc. of each solution in separate test tubes and add to each a solution of sodium hydroxide, drop by drop, until a precipitate appears. What is the precipitate in each case? Write the equations.

Divide each precipitate into two portions and treat one portion of each with a dilute acid and the other portion of each with some more sodium hydroxide solution. In the latter cases the hydroxide of the amphoteric metal is acting as an

acid toward the stronger base, sodium hydroxide. Write the equations.

Also write formulas for compounds in which As, Cr, and Mn form the cations and compounds in which these metals are present in the anions. The two types of compounds of the seven metals should now be arranged in parallel columns for comparison.

(*d*) **Action with Oxidizing Agents.** — The relative ease of oxidation of the metals is, in general, the same as the order of the metals in the Displacement or Electromotive Series. Any apparent exception will probably be due to the nature of the oxidation product which, if insoluble, forms a protective coating on the metal and prevents further action.

From their relative positions in the Electromotive Series would you expect aluminum or zinc to be oxidized more readily by nitric acid? Try the experiment, using dilute nitric acid, and explain the result.

Determine whether a ferrous or a ferric salt is formed when iron is acted upon by the following:

1. Dilute hydrochloric acid.
2. Dilute nitric acid.
3. Very dilute nitric acid.

To do this, clean the rust from three small iron nails by immersing them for a moment in a small amount of concentrated hydrochloric acid. Wash the nails with water and place them separately in three test tubes. Add 5 cc. of dilute hydrochloric acid to one tube, 5 cc. of dilute nitric acid to another, and to the third test tube add a solution made by diluting 1 cc. of dilute nitric acid with 5 cc. of water. Allow the reactions to proceed for at least ten minutes. The test tube containing the nail and hydrochloric acid should be heated at intervals to accelerate the action; do not heat the other two tubes.

Meanwhile, obtain a solution of a ferrous salt and a solution of a ferric salt from the shelf. To each add sodium hydroxide and note the character of the precipitate in each case. Equations. Apply this test to determine whether a ferrous or a ferric salt is formed by the reactions occurring in the three test tubes. If ordinary dilute nitric acid is made by adding 2 l. of water to a liter of the concentrated acid, what is the strength

of your very dilute nitric acid relative to concentrated nitric acid?

Drop a few iron filings into a test tube which is moist, on the inside. The filings should adhere to the walls of the tube. Allow this preparation to stand in your desk until the next laboratory period, then examine it and answer the following questions. When iron oxidizes (rusts) in moist air does the product form a protective coating and prevent further action? Explain why iron which is to be exposed to the weather is usually coated with zinc (galvanized) or tin or is plated with nickel.

By the method already outlined determine whether the oxide formed on the iron filings is a ferrous or a ferric compound. The rust should be separated from the iron which has not oxidized by adding some dilute hydrochloric acid and shaking the tube to suspend the oxide particles. This suspension should then be decanted from the metal and heated to dissolve the oxide. This solution should now be tested to determine whether a ferrous or a ferric salt is present. Equations.

EXERCISE 66

PROPERTIES OF SODIUM

Textbook: 538-543; 222-225

Take a **dry** watch glass to the storeroom and obtain 3 small pieces of sodium metal to use in the following experiments. (*Caution:* The sodium should be handled with forceps and must not come in contact with moist fingers nor with water, except as directed.)

(a) **General.** — Clean the oil from one piece of sodium by touching it on all sides with a piece of filter paper. Trim off one side of the sodium with a knife or a sharp edge of glass in order to expose a fresh surface of the metal. Examine and describe the metal with respect to **hardness, color, luster, tendency to tarnish** (oxidize); note its **melting point** and **density** in the table accompanying Exercise 64.

(b) **Reaction with Water.** — Place 10 cc. of water in a small beaker, drop into it the piece of sodium examined in (a), and quickly cover the beaker with a glass plate. When the reaction is complete, test the solution with litmus paper and rub a little

of it between your fingers (?). Equation. Explain how you could convert this product into common salt, stating how you would determine when you had added the exact amount of the required reagent.

(c) **Changes on Exposure to Air.** — This experiment will be understood only if it is kept in mind that the atmosphere contains carbon dioxide and water vapor as well as oxygen and nitrogen.

Clean the oil from another piece of sodium and scrape off as much of the coating as you can. Place the clean metal in a **dry** beaker and cut or mash the sodium into thin pieces in order to present a large surface to the air. Leave this exposed in your desk, notice it from time to time, and explain the following changes which will probably occur during the week:

1. The preparation becomes a solution. Test it with litmus and by touch. What is present? How did it obtain water? What is the technical name of this phenomenon?

2. It becomes crystalline. Test a portion with a dilute acid and explain the effervescence. What is the complete formula of this crystalline substance? What further change may occur?

(d) **Preparation of Sodium Peroxide.** — Place a plug of glass wool or cotton in a drying bulb, fill the bulb with granular calcium chloride to within 5 cm. of the end, and plug this end also with glass wool or cotton. Set up the apparatus as shown in Figure 24, placing a cleaned piece of sodium in a small porcelain boat in the ignition tube. By means of a

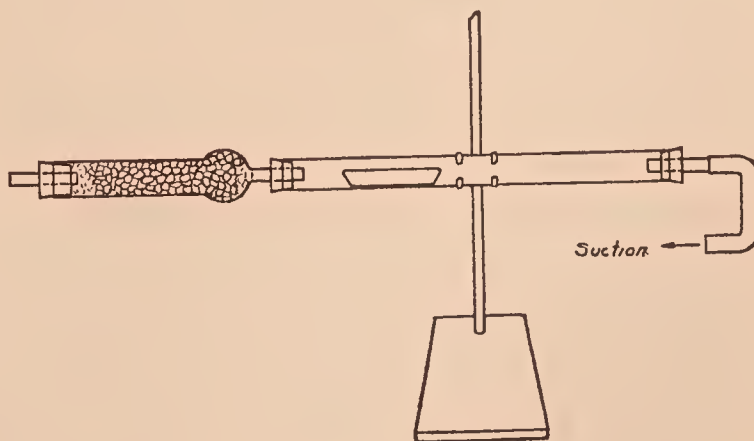


FIG. 24

suction attachment draw a slow stream of air through the drying bulb and over the sodium, which should be carefully heated until it melts. If no suction is available, air may be blown through from the other end. The molten sodium should take fire in the stream of air. Remove the gas flame as soon as the sodium begins to glow. When the reaction is complete allow the tube to cool, then remove and examine the product.

To 1 cc. of dilute sulfuric acid add 10 cc. of water, pour this into a test tube, and to this add some of the product obtained by burning sodium. Test the resulting solution for hydrogen peroxide, using 2 cc. of ether, 2 drops of a solution of potassium dichromate and shaking (?). What does this prove as to the product formed when sodium is burned? Suggest some reasons why it is advisable to dry the air before passing it over the sodium. What is the action of water on the combustion product of sodium? Equation.

EXERCISE 67

PREPARATION AND PROPERTIES OF SODIUM HYDROXIDE

Textbook: 225-231

(a) **Preparation.** — Write the equation for a reaction by which you have previously produced sodium hydroxide.

Weigh out 15 g. of sodium carbonate on the laboratory scales and calculate the weight of quicklime needed to react with this weight of material to produce sodium hydroxide. To make this calculation it is necessary to know whether the sodium carbonate which you are using is anhydrous or a hydrate and, if the latter, whether it is the monohydrate or the decahydrate. Write the formulas for the three possibilities. Devise a way to determine whether or not the sodium carbonate is a hydrate. If carefully heated, the decahydrate will melt before decomposing and may thus be distinguished.

Having calculated the weight of calcium oxide needed, weigh out 25 per cent in excess of this amount to allow for impurities. Add 15 cc. of water to the lime in a small beaker and allow it to stand until the lime has slaked. This will require about 10 minutes. Equation. In the meantime the sodium carbonate should be dissolved in 150 cc. of water in a beaker and heated to boiling. Add more water to the slaked lime to make a milky suspension and pour about three-fourths of this into the sodium carbonate solution. Stir this well and continue to boil it for a few minutes. Equation.

Now allow the precipitate to settle and transfer about 1 cc. of the clear solution to a test tube by means of a pipette. Treat this with dilute hydrochloric acid and notice whether or not a

gas is evolved (?). If effervescence occurs, all the carbonate has not been precipitated and more of the lime must be added. Continue the treatment with milk of lime and the testing with hydrochloric acid until all the carbonate has been removed from solution as CaCO_3 . Allow the precipitate to settle and decant the clear solution through a filter paper into a clean flask or bottle. Bring all the precipitate on the filter paper, allow it to drain and preserve it for use in Exercise 69.

(b) **Tests.** — Test small portions of the filtrate with two different indicators to show that it contains the OH^- ion. Place two or three drops of the filtrate on a watch glass and evaporate to dryness. Examine and briefly describe solid sodium hydroxide. Obtain a platinum wire and heat one end of it in the Bunsen flame until the wire no longer colors the flame. Now touch the red-hot end of the wire to the substance on the watch glass and again place the end of the wire in the flame (?). Test two other sodium compounds from the side shelf in this manner (?). This characteristic flame color may be used as a test for sodium compounds.

(c) **Reaction with Aluminum.** — Place a piece of aluminum in a test tube and cover it with some of the solution of sodium hydroxide. Does any reaction occur? Should aluminum utensils be washed or scoured with materials containing a strong alkali? Heat the test tube and contents and determine the identity of the gas evolved. To do this it may be necessary to collect a test tube full of the gas by the displacement of water. Write the equation for the reaction noting that the same compound of aluminum is formed here as in Exercise 65 (c).

(d) **Effect on Textiles.** — In separate test tubes place a small piece of wool and a bit of cotton cloth. To each add 5 cc. of the solution of sodium hydroxide, heat both tubes and note the effect. State how you could distinguish between cotton and wool by means of a strong alkali.

Save the remainder of the sodium hydroxide solution for the next exercise.

(e) **Analysis of a Wool-Cotton Mixture.** — Obtain a piece of the mixed cloth about 8 cm. square and carefully weigh it. Place this in a beaker and cover it with a 20 per cent solution of KOH or NaOH . To prepare this solution dissolve 5 g. of the solid alkali in the calculated amount of water. Boil the solution

for about three minutes, or until the wool is all dissolved. Avoid spattering the hot alkali on your flesh or clothing. Remove the cotton residue, taking care that no detached threads are lost. Rinse thoroughly, make slightly acid with dilute HCl and rinse again. Press the moisture from the cotton and dry it at about 100°C . by placing it in a dry beaker or dish and heating it carefully to avoid scorching. Weigh the residue and calculate the percentage composition of the fabric.

EXERCISE 68

HYDROXIDES OF THE METALS

(a) **Insoluble and Amphoteric Hydroxides.** — Arrange 10 clean test tubes in the rack and in these place separately about 2 cc. of solutions containing the following ions: Cu^{++} , Mn^{++} , Fe^{++} , Fe^{+++} , Zn^{++} , Al^{+++} , Pb^{++} , Ni^{++} , Co^{++} , Cr^{+++} . To each of these tubes add about 1 cc. (10 drops) of the sodium hydroxide solution prepared in Exercise 60 or the similar solution on the shelf. Write the equations, carefully recording the color of the precipitate in each case.

Heat test tube Number 1, containing the copper compound, and explain the change which occurs. Carefully note the colors first obtained in tubes 2 and 3 (repeat if necessary) and explain the changes which occur in these two on standing several minutes.

Now add an excess of sodium hydroxide to each of the ten tubes, with shaking, and note whether or not the precipitate dissolves. If solution occurs, write the equation — recall Exercise 65 (c).

(b) **Unstable Hydroxides.** — In separate test tubes study as in (a) the action of sodium hydroxide on solutions of a silver salt, a mercurous salt, and a mercuric salt. Describe the precipitates. They are the oxides of the metals — recall the change in copper hydroxide when heated. Note the position in the Electromotive Series of the metals which do not form stable hydroxides.

(c) **Classification.** — The soluble hydroxides are compounds of the metals high in the Electromotive Series. The solubility of these hydroxides in water at ordinary temperature is in the

same order as the activity of the metals. That is, KOH is the most soluble of the common hydroxides; ammonium hydroxide falls in third place. From these facts and your results in parts (a) and (b) of this exercise, tabulate the hydroxides in your notebook as follows:

SOLUBLE HYDROXIDES	HYDROXIDES PRECIPITATED BY NaOH, <i>insoluble</i> IN EXCESS	HYDROXIDES PRECIPITATED BY NaOH, <i>soluble</i> IN EXCESS (AMPHOTERIC)	OXIDES PRECIPITATED BY NaOH, HYDROXIDES UNSTABLE
Most soluble	Formula, Color	Formula, Color	Formula, Color
1.			
2.			
3.			
4.			
5.			
6.			
Least soluble			

EXERCISE 69

SODIUM BICARBONATE AND SODIUM CARBONATE

Textbook: 543-549

(a) **The Ammonia-Soda or Solvay Process.** — Saturate 20 cc. of concentrated ammonium hydroxide with sodium chloride in a test tube by adding an excess of the salt, shaking the mixture vigorously at intervals for some time, and then filtering out the undissolved solid. Catch the filtrate in a test tube and bubble carbon dioxide through it by means of a delivery tube reaching nearly to the bottom of the test tube. The carbon dioxide may be prepared by treating the precipitated calcium carbonate saved from Exercise 67 (a) with dilute hydrochloric acid in a generator as shown in Figure 25. The precipitate from 67 (a), if dry, should be broken into small lumps and sufficient water should be added to the generator to cover the end of the thistle tube. Add dilute hydrochloric acid through the thistle tube, a little at a time, as required to keep a slow stream of gas bubbling through the solution in the test tube. Continue the experiment

until a considerable amount of precipitate appears in the test tube. If this does not occur before the calcium carbonate in the generator is exhausted, the latter may be replenished with marble chips. If solid carbon dioxide, "dry ice," is available it may be added directly to the solution to produce the bicarbonate.

Filter the precipitate obtained in the test tube, spread out the filter and allow it to dry until it has lost the odor of ammonia.

The drying may be hastened by placing the paper in a dish on a water bath. Examine and describe the product, testing it for sodium and for the carbonate radical.

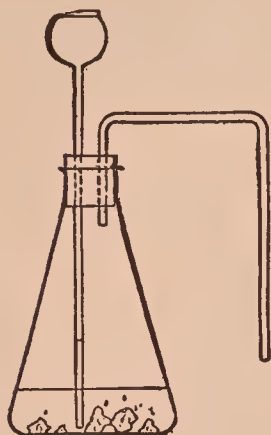


FIG. 25

In writing the equations for the formation of the precipitate it may be assumed that carbon dioxide reacts with ammonium hydroxide to form ammonium bicarbonate. This product may then react by double decomposition with sodium chloride precipitating sodium bicarbonate which is the least soluble of the substances involved.

(b) **Preparation of Sodium Carbonate.** — In a dry test tube place about 2 g. of sodium bicarbonate and fit the test tube with a stopper bearing a delivery tube bent to pass downward when the test tube is clamped in an inclined position. Arrange the lower end of the delivery tube to dip into 5 cc. of limewater in a test tube and then heat the sodium bicarbonate. What evidence have you as to two of the products of the decomposition of sodium bicarbonate? Equation. Is this reaction reversible? State exactly how you would proceed in converting sodium carbonate into sodium bicarbonate and write the equation.

(c) **Effects with Indicators.** — Dissolve about 0.5 g. of sodium bicarbonate in 20 cc. of water, divide the solution equally between two test tubes, add 2 drops of phenolphthalein to one and 2 drops of methyl orange to the other. From a pipette add dilute hydrochloric, drop by drop, to each tube until the indicator changes, noting the number of drops of acid required in each case. Does sodium bicarbonate affect one indicator more than the other? Does this show that various indicators change color at a slightly different H-ion concentration?

Dissolve about 4 g. of sodium carbonate in a sufficient quantity of water to make 100 cc. of solution. Place this solution in a clean flask or bottle. In another flask place 20 cc. of dilute hydrochloric acid and 220 cc. of water and mix well.

If ordinary dilute hydrochloric acid has a strength which is 6N, what is the normality of the acid solution which you have just prepared?

Obtain a burette, rinse it with some of the acid you have prepared and fill it with this acid. Using 25-cc. portions of the sodium carbonate solution for each experiment, titrate the sodium carbonate solution with the acid solution, using methyl orange as the indicator in one sample and phenolphthalein as the indicator in a separate sample (?).

Repeat the titration with each indicator, this time heating each 25-cc. sample of sodium carbonate solution to boiling and continuing to boil it while making the titration until the color change is permanent. Compare the results obtained in the four titrations and draw conclusions as to the reaction which occurred in each case. Equations.

EXERCISE 70

PREPARATION OF POTASSIUM NITRATE

Textbook: 554-555

Weigh out 25 g. of sodium nitrate and the amount of potassium chloride required to react with it. Place these two salts in a beaker, add 50 cc. of water and heat to boiling. The mixture should be stirred occasionally to hasten the process of solution. Filter the hot solution and allow the filtrate to cool. If desired, it may be placed in a beaker of cold water which should be renewed as it becomes warm.

From the solubility data in the accompanying table plot the solubility curves for the four salts over the temperature range 0° – 100° C. These four curves should appear on a single chart (see illustration in Exercise 64) with Temperature on the horizontal axis and Solubility (g. per 100 g. water) on the vertical axis.

SOLUBILITY IN 100 PARTS OF WATER

TEMPERATURE	0°	20°	40°	60°	80°	100°
KCl	28.5	34.7	40.1	45.5	51.	56.6
KNO ₃	13.3	31.2	64.	111.	172.	247.
NaCl	35.7	36.	36.6	37.3	38.5	39.8
NaNO ₃	72.9	87.5	102.	122.	148.	180.

When the solution has cooled to about 20° and crystals have formed, decant the mother liquor into another beaker, completely draining the crystals by inverting the beaker containing them over the other beaker as in Figure 26.

According to the solubility curves you have drawn, which one of the four salts must these crystals be? Calculate the weight of this salt you would expect to obtain at 20° from the amounts of solutes and solvent you have used. Examine and describe these crystals as to form (drawing) and taste. In reference below these will be called "A" crystals. Would these crystals be as pure if the solution had been cooled to 0° ? Why?

Evaporate the mother liquor to about one-half its volume, stirring it to prevent "bumping," and note the formation of

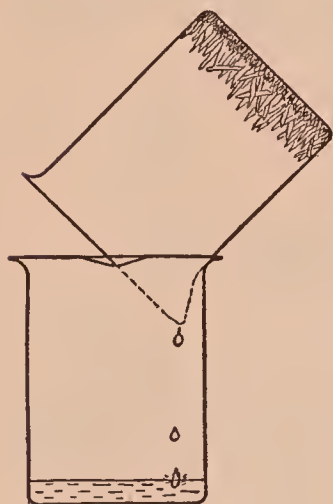


FIG. 26

crystals in the hot solution. These will be referred to as "B" crystals. Immediately decant the hot solution into another beaker and allow it to cool. Drain the crystals as before. Wash the "B" crystals by decantation using about 5 cc. of hot distilled water. Examine and describe these crystals as to form (drawing) and taste. Make a flame test with some of these crystals (?). Dissolve a few of these "B" crystals in water and test half of the solution for a chloride (?).

Try the brown-ring test for a nitrate on the other half of the solution to determine whether or not the crystals have been sufficiently washed.

When the hot solution has cooled, another crop of "A" crystals will be obtained. Drain these as before and combine them with the first crop. Dissolve all of the "A" crystals by heating them with a small amount of distilled water, 8 to 10 cc., and allow them to recrystallize. This process of recrystallization tends to purify the crystals. When the solution has cooled decant the liquid and bring the crystals into a filter paper fitted to a funnel. Allow them to drain and rinse them twice with 1-cc. portions of distilled water.

Dissolve a few of the crystals in water and make the nitrate and chloride tests on separate portions (?). If the chloride test is positive explain how you could further purify the crystals. Allow the crystals to dry on the filter paper and save them for Exercise 71.

EXERCISE 71

USES OF SOME POTASSIUM COMPOUNDS

Textbook: 556; 549–550

(a) **Preparation of Fuse Paper.** — Using the crystals of potassium nitrate prepared in Exercise 70 make a saturated solution at room temperature by shaking an excess of the salt in a test tube with 10 cc. of water. Soak a piece of filter paper and a short piece of wrapping cord in this solution and allow them to dry in the air. Heat a piece of glass tubing, draw it to a capillary and, using this as a pipette, trace a continuous design with the solution on a piece of filter paper starting at the edge of the paper. Mark the starting point with a pencil and when the paper has dried ignite it at this point only. A flameless combustion should travel around the design. What purpose does the potassium nitrate serve? When the string you have prepared is dry, ignite it and compare its rate of burning with that of an untreated piece. Twist pieces of the prepared filter paper into fuses for use as directed below.

(b) **Potassium Bitartrate — Baking Powder.** — Using the remainder of the potassium nitrate solution from (a) warm it to about 40° C. and saturate it at this temperature. To 5 cc. of this solution add an equal volume of a solution of tartaric acid, mix well and allow the tube to stand or hasten the crystallization by cooling with water. The precipitate is potassium acid tartrate, $\text{KHC}_4\text{H}_4\text{O}_6$, which is only slightly soluble. It is called “cream of tartar.” Write the equation. Filter out the precipitate, transfer it to a porcelain dish and apply heat, gently at first to dry it, then strongly. The tendency to char when heated or when treated with concentrated H_2SO_4 is characteristic of the salts of organic acids. When the residue is cool treat it with a dilute acid (?). From this test and from the appearance of the residue try to formulate the probable equation for the decomposition of $\text{KHC}_4\text{H}_4\text{O}_6$ by heat.

Calculate the weight of sodium bicarbonate required to react with 1 g. of cream of tartar to produce $\text{KNaC}_4\text{H}_4\text{O}_6$. Name this salt. Mix the required amounts of the two dry substances with a gram of starch. This mixture represents the more expensive class of baking powders. Divide the mixture between two dry test tubes, to one portion add cold water and to the other add

boiling water (?). Write the equation. Why did the reaction not occur when the salts were dry? Why is starch present in commercial baking powders?

(c) **Bengal Fires.** — In a clean dry mortar grind 5 g. of potassium chlorate to a fine powder. Pour this on a piece of paper, putting aside a small spoonful for (d) below. Wipe out the mortar and pulverize 1 g. of strontium nitrate or chloride. Add this to the potassium chlorate and mix these two powders on a piece of paper with 1 g. of “flowers of sulfur.” Mix thoroughly, using a spatula or the fingers, but do not grind together. (*Caution:* Do not grind sulfur or any other combustible material with potassium chlorate as a dangerous explosion will result.) Pour half of the mixture into a conical pile on a piece of asbestos and place a piece of fuse paper, at least an inch long, in the top of the pile. Light the end of the fuse paper and stand at a safe distance. What is the probable reaction between sulfur and potassium chlorate? The salts of strontium give a red color to flames. Salts of other metals could be substituted to give other colors.

(d) **Flashlight Powder.** — Mix the spoonful of powdered KClO_3 saved from (c) above with about an equal amount of magnesium powder. **Do not grind together.** Place this mixture in a pile on a piece of asbestos and ignite it using a fairly long piece of fuse paper. What is the application of this light? Why?

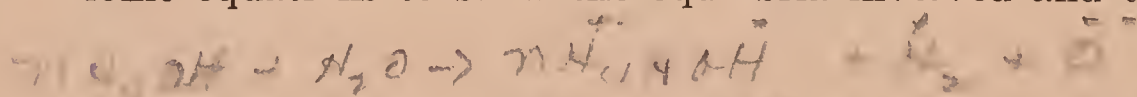
EXERCISE 72

AMMONIUM COMPOUNDS

Textbook: 558–561

The ammonium compounds closely resemble the corresponding compounds of potassium in crystal form, general appearance and solubility (as shown in Exercise 73). A few distinctive properties are outlined below.

(a) **Ammonium Hydroxide — Repression of Ionization.** — Dilute 2 cc. of ammonium hydroxide with water to 20 cc., add a few drops of phenolphthalein solution and divide the colored solution between two test tubes. Save one portion as a standard and to the other add 5 g. of solid ammonium chloride and shake the tube until the salt is dissolved. Explain the result, writing ionic equations to show the equilibria involved and the effect



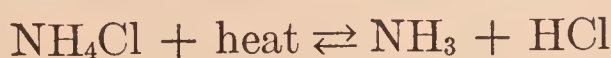
of the common ion. Could a similar result be obtained using the corresponding sodium or potassium compounds instead of NH_4OH and NH_4Cl ? Why?

To 5 cc. of a solution of a magnesium salt add 2 cc. of ammonium hydroxide (?). Equation. Repeat the experiment using ammonium hydroxide to which an equal volume of ammonium chloride solution has been added (?). Explain the different results. Add some ammonium chloride, either solid or in solution, to the test tube containing the precipitate and shake it (?).

(b) **Ammonia Complexes.** — Precipitate a small amount of silver chloride in a test tube and dissolve the precipitate by adding ammonium hydroxide. A soluble complex is formed having the composition $\text{Ag}(\text{NH}_3)_2\text{Cl}$. Test the effect of dilute nitric acid on this solution (?). Write the equations. Ammonia forms similar complexes with some other metals such as copper, zinc, cobalt, nickel. The number of molecules of ammonia contained in the complex ion is usually twice the maximum valence of the metal.

To a solution of copper sulfate add ammonium hydroxide slowly with shaking until the precipitate which forms is redissolved. The color of this complex is sometimes used as a test for copper. Write the formula for the copper-ammonia complex.

(c) **Removal of Ammonium Salts.** — In an analysis it is often necessary to remove ammonium salts as these interfere with certain tests. In a small, dry Pyrex beaker place a "pinch" of ammonium chloride. Heat the beaker on a wire gauze until all the salt has volatilized from the bottom of the beaker (?). The decomposition of this salt may be represented:



Does this indicate that a sublimate of the salt will condense on the cooler portions of the beaker? A reversible decomposition is called **dissociation**. When produced by heat, as in this case, it is called thermal dissociation. What name is given to the dissociation which occurs when an acid, base, or salt is dissolved in water?

Clean the beaker and repeat the experiment using ammonium nitrate instead of ammonium chloride (?). Write the equation for the thermal decomposition of this salt. Is the reaction reversible? Should we call this dissociation? With what reagent

might the NH_4Cl have been treated before heating to effect a complete removal of ammonium salt? Show by means of an equilibrium equation why your answer is true.

(d) **Test for the Ammonium Radical.** — Recall or test the action of a strong alkali on ammonium salts and write an equilibrium equation to show why this reaction may be used to identify ammonium salts. State several means by which you can recognize ammonia gas.

EXERCISE 73

TESTS FOR POTASSIUM, SODIUM, AMMONIUM SALTS

Textbook: 561–563

Most salts of these cations are quite soluble. A few, however, are only slightly soluble and may be precipitated if present in sufficient concentration — recall $\text{KHC}_4\text{H}_4\text{O}_6$ as a slightly soluble potassium salt.

(a) **Perchlorates.** — In separate test tubes place 2-cc. portions of solutions of a potassium salt, a sodium salt, and an ammonium salt. To each solution add a few drops of perchloric acid (storeroom) and describe the results. Equations. How could you separate sodium and potassium from a mixture of their salts? Alcohol ($\text{C}_2\text{H}_5\text{OH}$) is usually added to decrease the solubility of potassium perchlorate and make the separation more complete. Why is it necessary to remove ammonium salts before testing for potassium with perchloric acid? Recall Exercise 72 (c) for the method.

(b) **Fluosilicates.** — Again using 2-cc. portions of solutions of the three salts as in (a) add an equal volume of alcohol (store-room) and then a few drops of fluosilicic acid, H_2SiF_6 (store-room) to each test tube. Allow the tubes to stand for a few minutes and carefully note the differences in the nature of the precipitates if more than one is obtained. Equations.

(c) **Cobaltinitrites.** — To solutions of potassium, sodium and ammonium salts add a solution of sodium cobaltinitrite, $\text{Na}_3\text{Co}(\text{NO}_2)_6$. This reagent is prepared as follows: Dissolve 1 g. of sodium nitrite in 5 cc. of water, add 2 cc. of cobalt nitrate solution and 1 cc. of acetic acid. Shake this mixture, allow it to stand a few minutes, and then dilute it with an equal volume of water. Use this to make the tests as directed above

and describe the results. Equations. Is it necessary to remove ammonium salts before testing for potassium with this reagent?

(d) **Flame Tests.** — Clean a platinum wire by repeatedly dipping it in some HCl solution in a watch glass and heating it in the non-luminous flame until it gives practically no color. Test several potassium salts, both solids and solutions, with the platinum wire in the non-luminous flame (?). Recall or repeat the flame test for sodium. Now mix solutions of KCl and NaCl in equal parts and make a flame test with this mixture. Which metal gives the predominant color? Is the test for the other metal entirely masked? Now repeat the flame test with the mixture, this time viewing the flame through a piece of cobalt glass (?).

Clean the platinum wire and make a flame test with an ammonium salt. This dull coloration is given by many substances and hence there is no characteristic flame for ammonium salts. Because of the wide distribution of sodium salts be careful to distinguish the brilliant and persistent coloration given the flame by considerable amounts of sodium from the flash given by a trace such as may be obtained by drawing a clean platinum wire between the fingers.

Tabulate the tests for these three cations somewhat as follows:

	HClO ₄	H ₂ SiF ₆	Na ₃ Co(NO ₂) ₆	FLAME
Na K NH ₄				

(e) **Analysis of Unknown No. 1.** — This unknown will consist of a salt of sodium, potassium or ammonium or a mixture of any two or all three. Report only the cations in this analysis.

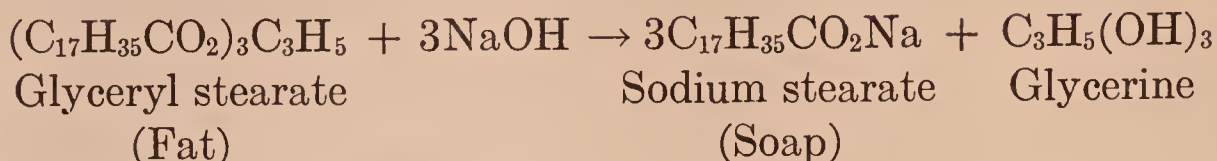
EXERCISE 74

SOAP

Textbook: 499–500

(a) **Preparation of Soap.** — “Soap” is the common name for the sodium salts of the fatty acids, usually palmitic, stearic or oleic. It is made by treating a fat with an alkali, the composi-

tion of the soap depending upon the nature of the fat which is used, for example:



This type of reaction is called **saponification**. Glyceryl stearate is the principal fat in beef tallow. In olive oil the fat is glyceryl oleate. Lard is a mixture of the glyceryl esters (organic salts) of the three acids mentioned above. What then is the composition of Castile soap made from olive oil? Of soap made from hog fat?

Weigh 15 g. of lard or other fat into a small beaker; allow 5 g. of sodium hydroxide to dissolve in 5 cc. of water, add this to the fat, and place this beaker in another beaker of boiling water. Heat the mixture in this way for about an hour, stirring occasionally and adding a small quantity of water as needed. The contents of the beaker should stiffen. Remove a small portion of the mass on a stirring rod and test it with a drop of phenolphthalein. Should a good toilet soap give this test? Why? What other impurity is present in this soap? Soap is fairly soluble in water but is insoluble in a saturated solution of sodium chloride. Your product should be purified as follows: Remove the mass to a larger beaker, add 100 cc. of distilled water and boil this mixture, breaking up any lumps with a spatula. Saturate the water with finely powdered sodium chloride while heating and stirring the solution. Allow the mixture to cool and remove the cake of soap. It should be pressed into a filter paper fitted to a funnel and allowed to dry there. Test its ability to form a lather by shaking a small quantity with 10 cc. of distilled water. Finally weigh your product and submit it to your instructor for inspection.

(b) **Detergent Action.** — The detergent (cleansing) action of soap is dependent upon its property of taking grease and dirt into colloidal suspension and thus mechanically removing them by *emulsification* and *peptization*.

Prepare a 1 per cent soap solution by shaking 2 g. of soap with the calculated volume of distilled water in a flask, heating it to complete the solution. Use this solution throughout this exercise and save the remainder for Exercises 75 and 77.

In separate test tubes place 20 cc. of water and 20 cc. of

soap solution. To each tube add 1 g. of animal charcoal and shake the tubes vigorously. Compare the amounts of solid held in suspension after the tubes have stood for several minutes. The action of soap in removing grease and oil is indicated in Exercise 75.

(c) **Action with Ca^{++} , Mg^{++} , K^{+} , etc.** — Add some soap solution to a solution of calcium chloride and filter off the precipitate. What is it? Assuming the soap to be sodium stearate write the equation. Shake some of the precipitate with water. Does it dissolve? Lather? Does it have the cleansing property of a soap?

Add soap solution to solutions of KCl , MgCl_2 , FeCl_3 and any other chlorides. What other metal might be substituted for sodium in soap? Soaps of this metal do not harden; they are liquids.

Add 2 cc. of dilute sulfuric acid to 10 cc. of soap solution (?). Equation. Shake the tube vigorously to coagulate the precipitate. What is it? Will it dissolve when boiled with a solution of NaOH ? Equation.

EXERCISE 75

EMULSIONS

Textbook: 148

A dispersion of droplets of one liquid in another liquid is called an emulsion. It is, of course, necessary that the two liquids be mutually insoluble (immiscible). It is that type of colloidal solution in which both components are **liquids**. A familiar example of an emulsion is that formed from water and an oil.

(a) **Preparation of an Emulsion.** — Pour 10 cc. of kerosene and 10 cc. of water into a test tube. Likewise mix 10 cc. of kerosene with 10 cc. of soap solution. Shake both tubes vigorously and compare them after they have stood a few moments (?). Are oil and water miscible? Can a stable emulsion be prepared by shaking oil and water together? Explain in your own words what happens when an emulsion “breaks” or separates into layers of its two components. An emulsifying agent retards or prevents breaking because it forms a film around the dispersed droplets and keeps them from contact and

union with each other. Does your experiment indicate that soap is an emulsifying agent? Save the tubes and contents for (b).

Make a solution of albumin (white of egg) by shaking a small quantity of dried albumin with 10 cc. of water in a test tube. To this solution add an oil, a few drops at a time, shaking the tube vigorously, until 5 cc. of the oil have been added (?).

An emulsion used for salad dressing is made by stirring together vinegar (dilute acetic acid), olive oil (cottonseed or corn oil may be substituted), and eggs. What is the familiar name for this emulsion? What is the emulsifying agent in this case?

(b) **Types of Emulsion.** — Make a simple sketch to represent a highly magnified section of an emulsion. Label the droplets “internal phase” or “dispersed phase” and the surrounding liquid “external phase,” “continuous phase,” or “dispersion medium.” It is obvious that, theoretically, in the case of oil and water either may be the internal phase. That is, we may have oil droplets dispersed in water or water dispersed in oil. It is easy to determine which condition is actual by adding more of one of the components to the emulsion. If the external phase is added it will mix readily; if the internal phase is added it will not mix.

Shake the tube reserved from (a) containing kerosene and soap solution and divide the emulsion into two nearly equal portions. To the test tube containing one portion add water and to the test tube containing the other about the same amount of kerosene. Cover each tube with a thumb and invert it once to mix the materials but do not shake (?). Is water or oil the external phase in this emulsion?

Prepare some magnesium stearate by adding 10 cc. of a solution of MgCl_2 or MgSO_4 to 10 cc. of soap solution. Filter, wash, and partially dry the precipitate by pressing it between filter paper. Add it to the oil-water mixture saved from (a) and shake vigorously (?).

Divide this material into two equal portions and determine as before the identity of the internal and external phases. Is magnesium stearate an emulsifying agent? How does the action of magnesium stearate differ from that of sodium stearate?

EXERCISE 76

CUPROUS COMPOUNDS

Textbook: 575-577: 503-506

(a) **Cuprous Chloride.**—Dissolve 3g. of cupric chloride in 15cc. of water, add 2 g. of copper turnings and 3 cc. of concentrated hydrochloric acid. Boil the solution gently for several minutes or until the green tint is no longer perceptible. Equation.

Boil 20 cc. of water in a test tube to remove the dissolved oxygen, pour about 1 cc. of the cuprous chloride solution into this water (?). What is the precipitate? If a blue color appears in the solution was the reduction of cupric chloride complete?

To 5 cc. of the cuprous chloride solution add a solution of sodium hydroxide until the mixture is alkaline (?). Equation. Compare this result with that obtained when sodium hydroxide solution is added to a solution of a cupric salt.

Pour the remainder of the cuprous chloride solution into a large volume of water (?). Equation. Decant or filter the solution and test the solubility of portions of the precipitate in dilute hydrochloric acid and in ammonium hydroxide. Expose a portion of the precipitate to the air on a piece of filter paper (?). Explain your results.

(b) **Cuprous Iodide.** — Dilute 3 cc. of copper sulfate solution with an equal volume of water and add 3 cc. of potassium iodide solution (?). Filter out the precipitate and account for the color of the filtrate (?). Dilute some of the filtrate and test it with starch solution (?). Write the equation for the action of a cupric salt with potassium iodide.

(c) **Fehling's Solution.** — Dissolve 3.5 g. of copper sulfate crystals in water and dilute the solution to 50 cc. Place this in a small bottle or flask and label it "Solution A." Dissolve 5 g. of solid sodium hydroxide and 12.5 g. of Rochelle salt (potassium sodium tartrate, $\text{KNaC}_4\text{H}_4\text{O}_6 \cdot 4\text{H}_2\text{O}$) in water together and dilute this solution to 50 cc. Bottle this and label it "Solution B."

Mix 5 cc. of Solution A with 5 cc. of Solution B (?). What would have been the result in the absence of the Rochelle salt? To this mixture add 1 cc. of glucose solution and boil the solution noting all changes. What is the precipitate? What was

the action of glucose on the cupric salt? This reaction is used in detecting and estimating glucose and other sugars of the same type.

Repeat the Fehling test using a solution of cane sugar instead of glucose (?). "Invert" some cane sugar by boiling 10 cc. of its solution with a few drops of concentrated hydrochloric acid for several minutes. Carefully neutralize the acid with sodium hydroxide solution and test this invert sugar with Fehling's solution as above — that is, mix 5 cc. of Solution A and 5 cc. of Solution B; add 2 or 3 cc. of the solution to be tested and boil several minutes (?). Hydrogen ions catalyze the hydrolysis of cane sugar ($C_{12}H_{22}O_{11}$) to form glucose and fructose, both of which have the formula $C_6H_{12}O_6$. Write the equation for this "inversion."

Prepare a starch solution by moistening a pinch of starch with water and adding boiling water to this. Test some of this starch solution with Fehling's solution as directed above (?). To 2 cc. of thin starch solution in a test tube add about 5 cc. of saliva, warm the tube slightly by immersing it in hot water and shake it for several minutes. Test the resulting solution with Fehling's solution (?). The first stage in the digestion of starch is its conversion to sugars through the agency of the enzyme **diastase** in the saliva.

(d) **Testing Common Substances for Glucose.** — If any of the following substances are available test them for glucose (or similar sugars): Table syrups such as corn syrup, molasses, maple syrup; fruit juices — orange, apple, raisin, etc.; jams and jellies; candies. Use a clear solution of the material for the test.

EXERCISE 77

PREPARATION OF SOME CUPRIC COMPOUNDS

Textbook: 575–578

(a) **$CuSO_4 \cdot 5H_2O$.** — Weigh 5 g. of copper, place this in a clean beaker, and add 10 cc. of concentrated sulfuric acid, 20 cc. of dilute nitric acid and 20 cc. of water. Heat this mixture over a low flame in the hood until the copper has all dissolved, adding more dilute nitric acid if necessary. Add more water if crystals

begin to form before all the copper has dissolved. While this is in progress proceed with (b) of this exercise. When solution is complete, filter the liquid if it is not clear, catch the filtrate in a clean beaker and evaporate this solution until crystals begin to form. Place the beaker in cold water and stir the liquid until crystallization is complete. Decant the liquid from the crystals, evaporate the mother liquor and crystallize it as before. Combine the two crops of crystals, drain them and dissolve them in the minimum amount of boiling water in a small beaker. Cover this solution with a watch glass and set it in your desk for a day or more to recrystallize.

Decant the liquid from this last crop of crystals, rinse them with a small amount of distilled water, and dry them between filter papers. Weigh the crystals and calculate your per cent yield from the weight of copper used in the experiment. Select a good crystal, rinse it again, dissolve it in water and test small portions for: (1) Sulfate; (2) Nitrate (as impurity); (3) Copper (use the ammonium hydroxide test and also test with a solution of potassium ferrocyanide). Copper gives a chocolate-colored precipitate of cupric ferrocyanide with this reagent; (4) Also test a small dry crystal of your product for water of hydration. What is the color of the anhydrous salt?

Assuming that nitric acid acted as the oxidizing agent, being reduced to nitric oxide, write the equation for the production of copper sulfate in this experiment.

Ask your instructor to inspect the copper sulfate crystals and your record for the experiment. Save the crystals for (c).

(b) $\text{Cu}(\text{NH}_3)_4\text{SO}_4 \cdot \text{H}_2\text{O}$. — Grind 10 g. of copper sulfate to a fine powder, place this in a small flask and add 20 cc. of concentrated ammonia solution. Shake the flask for some time and then warm gently to complete the solution of the solid. Transfer the solution to a beaker, cover it and place it in your desk to crystallize. Filter off the crystals and dry them very carefully with filter paper. Submit the crystals to your instructor for inspection.

(c) $\text{CuSO}_4 \cdot (\text{NH}_4)_2\text{SO}_4 \cdot 6\text{H}_2\text{O}$. — Using the crystals of blue vitrol prepared in (a) or some from the reagent shelf, grind 10 g. to a fine powder. Calculate the weight of ammonium sulfate equivalent to the 10 g. of copper sulfate. Weigh out this amount of ammonium sulfate, grind it with the copper sulfate in the mortar, transfer the mixture to a small flask or

beaker and dissolve it in the smallest possible amount of boiling water. Acidify this solution with a few drops of dilute sulfuric acid and immediately filter it while hot into a small beaker. Cover the beaker and set it in your desk for a day or more to crystallize.

Drain, rinse and dry the crystals, and submit them to your instructor for inspection. Compare the crystals of the three substances you have prepared in this exercise and state how you could distinguish them from each other, either by appearance or chemical tests. Clearly distinguish by **name** between the compound prepared in (b) and that prepared in (c) of this exercise (?).

EXERCISE 78

METALLIC COUPLES

Textbook: 578

(a) **Displacement.** — Place a piece of zinc and a piece of copper (not in contact) in the bottom of a beaker and cover them with dilute sulfuric acid. What reaction occurs?

Bring the two metals into contact (?). Is the rate of the reaction changed? Explain the evolution of gas from the surface of the copper. How could you determine whether or not any copper is dissolved? Remove the piece of copper and try the effect of touching the zinc under the acid with a platinum wire (?). Is any energy evolved in this reaction? In what form does it appear? Explain how the addition of copper sulfate accelerates the production of hydrogen from zinc and sulfuric acid.

Pour a few drops of lead nitrate solution into a watch glass and place a piece of zinc in this for a few moments (?). Equation. The zinc should be only partially submerged in this solution. Amalgamate a piece of zinc by covering it with a solution of a mercuric salt for a few moments. Equation. Place these two pieces of zinc in separate test tubes and in a third tube place an ordinary piece of zinc. Cover each preparation with dilute sulfuric acid, observe and record what happens. In a zinc amalgam the mercury dissolves in the zinc exposing a surface which is equivalent to pure zinc. Ordinary zinc contains other metals as impurities. Does pure or impure zinc react more readily with an acid? Explain.

From the standpoint of the E.M.F. series, which of two metals in contact will dissolve first in an acid? From the surface of which metal is most of the hydrogen liberated? To answer this question cover a piece of amalgamated zinc (pure zinc) with dilute sulfuric acid and touch the zinc under the liquid with a platinum wire (?).

(b) **Action of Metals with Water.** — Boil some water in a beaker to remove the dissolved air. In separate test tubes place a piece of magnesium ribbon, a piece of aluminum and a piece of zinc. Add some of the boiling-hot water to each of these samples and observe whether or not any hydrogen is produced. Now add a few drops of a solution of a mercuric salt to the test tubes containing magnesium and aluminum and some copper sulfate solution to the test tube containing zinc. Again heat the water to the boiling point and notice if any of the metals displace hydrogen. Explain.

EXERCISE 79

ELECTROCHEMICAL ACTION

Textbook: 578–581

(Two students may work together on this exercise)

(a) **Electricity from Chemical Action.** — Obtain two zinc electrodes and two copper electrodes from the storeroom. Place one zinc electrode and one copper electrode in a beaker and hold these in place and apart by slipping a smaller beaker within the other. Mix some dilute sulfuric acid with twice its volume of water and pour some of this solution into the outside beaker. Add water to the inner beaker if necessary to give it weight. Is any action visible at either electrode? Connect the free ends of the wires leading to the electrodes. State what happens at each electrode while the cell is thus short-circuited. Disconnect the wires to break the electrical circuit while the cell is not in use.

Amalgamate the other zinc electrode, rinse it, and set up a separate cell similar to the one just described. Observe this cell for action at the electrodes when the circuit is open and closed, and compare this cell with the one first prepared. What is the purpose of amalgamating the zinc electrode?

Connect the two cells in series by joining the copper electrode of one to the zinc electrode of the other. Determine the sign of each electrode (+ or -) by means of the following polarity tests: Add about 3 cc. of thin starch solution to 5 cc. of potassium iodide solution. Moisten a piece of filter paper with some of this mixture and save the remainder for (b). Touch the ends of the free wires to the moistened paper — the ends of the wires should be about 1 cm. apart. This test may also be performed by pouring some of the solution in a watch glass and holding the wires 1 cm. apart in the liquid (?). What causes the color? At which electrode does it appear? A similar polarity test may be made with a solution of sodium chloride containing a few drops of phenolphthalein. Test as before and explain the appearance of the color. At which electrode is the color seen in this case?

Determine the voltage (E.M.F.) of this battery of two cells by connecting its wires to the proper terminals of a voltmeter (?). Write an ionic equation to show the source of the electric current in these cells.

(b) **The Daniell Cell.** — Rinse the zinc and copper electrodes used in (a), amalgamate the other zinc electrode, and obtain

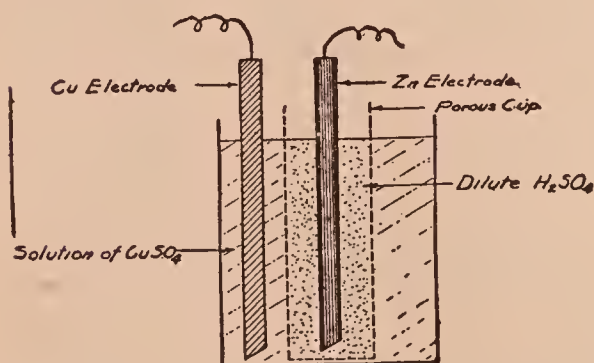


FIG. 27

two porous cups from the store-room. Assemble two cells as illustrated in Figure 27, placing dilute sulfuric acid in the porous cup and a saturated solution of copper sulfate in the beaker surrounding the porous cup. Is any chemical action visible? Is any action visible when the circuit is completed

by joining the wires from the electrodes? Make a polarity test as directed in (a). This test will be obtained more readily if the cells are first connected in series. Using the voltmeter as before, determine the E.M.F. of a single Daniell cell, the E.M.F. of the two cells connected **in series**, and the E.M.F. of the two cells connected **in parallel**. Compare these values (?). When the two cells are connected in series sufficient current may be obtained from them to cause a small flashlight bulb to glow.

(c) **Electrolysis.** — Dip the free ends of the wires from the Daniell cells (connected in series) into a solution of copper sul-

fate. Does electrolysis occur? What happens at each pole? What practical application is illustrated by this experiment?

Rinse the ends of the wires and dip them into a solution of sodium hydroxide. What occurs at each electrode in this solution? Explain how this experiment could be used as a polarity test. What would have been the products of this electrolysis if the electrodes had been platinum instead of copper?

Remove some of the liquid from one of the porous cups by means of a pipette and test it for zinc ions as follows: Make the solution alkaline with ammonium hydroxide and pass in hydrogen sulfide. Refer to Exercise 84 (d), first paragraph. A white precipitate, soluble in hydrochloric acid, is zinc sulfide. What occurs at each electrode of the Daniell cell when it is in operation? Express the net chemical change in the cell by means of an ionic equation and indicate the source of the electrical energy.

EXERCISE 80

SILVER

Textbook: 581-590

(a) **Displacement of Silver.** — Place a drop of metallic mercury in 5 cc. of silver nitrate solution in a test tube and allow this preparation to stand undisturbed until the next laboratory period. At that time describe the result and write the equation.

Mix 2 cc. of silver nitrate solution and 2 cc. of cupric sulfate solution in a test tube. Dilute this mixture with an equal volume of water and drop into it a piece of zinc (?). Describe the deposit which appears immediately and that which appears after a few minutes. Is the black deposit copper? To decide this drop a piece of zinc into a mixture of 2 cc. of silver nitrate and 6 cc. of water and compare the results (?). After the solution which contained both copper and silver has been in contact with the piece of zinc for at least 10 minutes, remove about 2 cc. by means of a pipette. Test separate portions of this for copper, see Exercise 77 (a), and for silver. Devise a test for silver based on the fact that silver chloride is insoluble in water and dilute acids but soluble in ammonium hydroxide. Is copper

or silver completely displaced from the solution? Leave the remainder of the solution in contact with the zinc until the next laboratory period and test the solution for copper, silver and zinc (?). Write the equations.

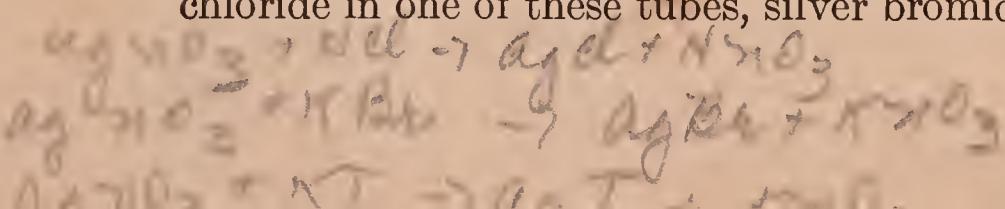
(b) **Effect of Light.** — Using silver nitrate solution as an ink, mark some design on a piece of filter paper. Allow this to dry and then warm it gently (?). The same effect is produced by longer exposure to light. Mark a towel or other cloth with silver nitrate solution and notice it after a day or two (?). The stain will not wash out. Can you suggest a use for this ink? Similar stains will be made on the skin by silver nitrate solution. Why is silver nitrate usually kept in a brown bottle? Note the stains at the mouth of the bottle of silver nitrate solution. What is this stain? How would you prove it?

Add 5 cc. of sodium chloride solution to 5 cc. of silver nitrate solution, filter quickly and transfer half of the precipitate to another filter paper. Immediately place one portion of the precipitate in the dark and expose the other portion to the light. Notice the changes which occur in the latter and finally compare it with the other portion which was left in the dark. Scrape the surface of the portion exposed to light (?). Has the action penetrated to a considerable depth? The effect of light is to make the silver salts more readily reducible to metallic silver. Explain the practical application of this fact. What is a “developer”? Mention one. What is a “fixing agent”? Mention the one commonly used.

(c) **Reactions of the Silver Ion.** — Add sodium hydroxide solution to 2 cc. of silver nitrate solution (?). Refer to Exercise 68 (b). Equation. What other cations give similar results?

Add ammonium hydroxide, drop by drop, to 1 cc. of silver nitrate solution (?). Write two equations to illustrate your observations. What molecules and ions are present in the solution called ammonium hydroxide? Show this by indicating the equilibria involved. Which ion or molecule is responsible for the first change noted in the reaction with silver nitrate? Which causes the second change? What complex ion is finally present? Refer to Exercise 72 (b).

Pour 2 cc. of silver nitrate solution into each of three test tubes and dilute each portion with about 5 cc. of distilled water. By adding solutions of the proper reagents precipitate silver chloride in one of these tubes, silver bromide in the second, and



silver iodide in the third. Divide each precipitate into two portions and test the solubility of the three silver halides in ammonium hydroxide and in a solution of sodium thiosulfate. Tabulate your results so that it is obvious how these three silver salts may be distinguished by color and by solubility.

(d) **Silvering a Mirror.** — Carefully clean a watch glass by washing it with a hot, 10 per cent solution of sodium hydroxide, rinse it first with tap water and finally with distilled water. Pour 4 cc. of silver nitrate solution into the watch glass and add ammonium hydroxide, drop by drop, until the precipitate which first forms has dissolved. Add about 3 cc. of a 10 per cent glucose solution to the solution in the watch glass and warm the mixture by placing the watch glass over a beaker of boiling water. Continue heating the water bath until a considerable deposit of silver is produced. What chemical property of glucose is utilized here? Recall its action in Exercise 76 (c).

Rinse the watch glass and dry it by pressing a clean cloth or filter paper against it. Examine the mirror from the under side of the watch glass. Write an equation to show how this deposit may be removed from the glass.

EXERCISE 81

REACTIONS OF CALCIUM, STRONTIUM AND BARIUM

Textbook: 597-617

(X) **Flame Tests.** — Clean a platinum wire as directed in Exercise 73 (d) and test a solution of calcium chloride in the flame (?). Repeat the test with strontium and barium salts, being careful to clean the wire after each test. The flame given by lithium salts is not easily distinguished from the strontium flame; copper compounds and boric acid give a flame color similar to that of barium salts. It is not easy to distinguish calcium, strontium and barium by means of the flame test when two or three of these metals are present. For this reason additional tests must be employed in detecting these cations.

(X) **Precipitation Tests.** — Perform the following tests separately on 5-cc. portions of solutions of calcium, strontium and

10 cc of water

GENERAL CHEMISTRY

barium salts. Describe the results, write the equations, and collect your data in a table similar to the form suggested below. Indicate the form and color of the precipitates.

1. Add a solution of sodium carbonate or ammonium carbonate (?).
2. Add a solution of ammonium oxalate (?).
3. Add a solution of potassium dichromate (?).
4. Add a saturated solution of calcium sulfate (?).

In test 4 note whether the precipitate forms quickly or only after warming and standing. From this observation draw a conclusion as to the relative solubility of the three sulfates. Of the carbonates and oxalates, the calcium salts are least soluble. Indicate the order of solubility for the sulfates, carbonates, oxalates and chromates of the alkaline earths, remembering that the strontium compound is always intermediate in solubility.

	CARBONATE	OXALATE	CHROMATE	SULFATE	FLAME
Calcium	milky	milky	amorphous yellow	white	red
Strontium	"	"	"	"	"
Barium	"	"	"	"	green

(c) **Separation.** — Starting with a solution containing Ca^{++} , Sr^{++} , and Ba^{++} (use chlorides or nitrates) separate the barium as a precipitate (?). Filter out the precipitate and divide the filtrate into two portions. In one portion precipitate strontium by adding a saturated solution of calcium sulfate and warming (?). To the other portion add a solution of ammonium sulfate, boil and filter. Test for calcium in this filtrate by adding a solution of ammonium oxalate (?).

(d) **Analysis of Unknown No. 2.** — This unknown will consist of a salt of calcium, strontium or barium or a mixture of any two or all three. Report only the cations in this analysis.

EXERCISE 82

HARD WATER

Textbook: 607-609

(a) **Soft Water.** — To ~~20 cc.~~ ^{10 cc. Tap} of distilled water in a test tube add soap solution, a few drops at a time from the 10-cc. graduate cylinder, shaking the tube vigorously, to determine the volume of soap solution required to give a collar of lather or suds which will last at least 10 seconds. Record this value for comparison below. (Repeat this test with tap water and determine if it has any degree of hardness by comparing the amount of soap needed to give a lather in this case with the value obtained for distilled water.)

(b) **Temporary Hardness.** — Generate carbon dioxide in a small flask, using marble chips and dilute sulfuric acid, and ~~pass~~ ^{omit} this gas into 20 cc. of limewater until the precipitate which forms at first has completely dissolved. What was the precipitate? What is now present in the solution? Equations.

Using 10 cc. of this solution determine the volume of soap solution required to give a lather (?). Boil the other portion of the solution for some time and note the change which occurs (?). Equation. Now make the soap test on this boiled solution and compare the volume of soap solution required with that used in the portion not boiled (?). What compounds cause temporary hardness in water? How may temporarily hard water be softened? Explain how it would be possible to soften such water by adding exactly the required amount of limewater? Equation.

(c) **Permanent Hardness.** — Determine the volume of soap solution required to give a lather with 20 cc. of a saturated solution of calcium sulfate solution and compare this value with that obtained for soft water (?). To 20 cc. of the calcium sulfate solution add a gram of solid sodium carbonate and mix well by shaking the tube (?). Equation. Make the soap test on this solution and compare with the values above (?). What has been accomplished by the use of sodium carbonate? Repeat the test using borax in place of sodium carbonate (?). What compounds may be present in water of permanent hardness? What is the practical distinction between the two types of hard water? Explain the white deposit formed in water kettles and boilers in limestone regions.

EXERCISE 83

MAGNESIUM

Textbook: 622-627

(a) **Properties of the Metal.** — Hold a piece of magnesium ribbon by one end with the forceps and heat it about 2 cm. from the other end by means of a lighted match. Is there any evidence of melting? Now heat the ribbon at the end to ignite it, using the Bunsen flame if necessary, and hold the burning ribbon over a small beaker to catch the solid product. Assuming that the burning magnesium combines to some extent with both of the principal components of the atmosphere, write two equations to show the products formed. Add about 2 cc. of water to the products in the beaker, heat the water to boiling and test the solution with litmus paper (?). Write the equations for the action of water upon the two combustion products. What use is made of the fact that the light from burning magnesium is rich in ultra-violet rays? Refer to Exercise 71 (d).

Write equations for the action of magnesium with the dilute solutions of the three common acids on the desk.

(b) **Some Magnesium Salts.** — Place a few crystals of magnesium chloride on a watch glass and expose it to the atmosphere for a day or more (?). Compare this result with the action of a calcium chloride under the same conditions.

Test concentrated solutions of magnesium chloride and magnesium sulfate with litmus and explain the result. To make this test, crystals of the salt may be placed on moistened litmus paper.

Place a gram of magnesium chloride crystals, $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$, in a porcelain crucible and heat it. Test the escaping vapor with moist litmus paper (?). Explain. Finally heat the crucible at a red heat until the vapor is no longer evolved. What is the residue? Equation. Mention a practical use of this substance.

Heat a small quantity of magnesite in a hard glass test tube and pass the gas which is driven off into some limewater (?). Equations. Heat the magnesite until the decomposition is complete, empty the residue into a small beaker and add

about 10 cc. of water. Boil the water and test the solution with litmus paper and a portion of it with phenolphthalein (?). What is present in the solution? Did all the residue dissolve? Suggest a reagent which will dissolve it readily, and test your idea. Equation.

(c) **Precipitation of Magnesium — Test.** — Refer to Exercise 72 (a), second paragraph, for the effect of ammonium salts in preventing the precipitation of magnesium hydroxide. Repeat the experiment if necessary.

To 5 cc. of a solution of magnesium salt add a solution of ammonium carbonate (?). Equation. To 5 cc. of the magnesium salt solution add an equal volume of ammonium chloride solution, mix by shaking the test tube, and then add ammonium carbonate solution (?). Explain.

Repeat the last paragraph using a solution of a calcium, strontium or barium salt in place of the magnesium salt (?). On this basis explain how you could separate the alkaline earth elements from magnesium.

To 5 cc. of a solution of a magnesium salt add an equal volume of ammonium chloride solution, then make the solution strongly alkaline with ammonium hydroxide and add a solution of sodium phosphate (?). Why was the ammonium chloride added? The precipitate is MgNH_4PO_4 . Describe its appearance, name it, and write the equation. This is used as a test for magnesium. State how you would use the test conversely for a phosphate. What should be the composition of "magnesia mixture" used in testing for phosphate? Filter out the precipitate, transfer some of it to a porcelain crucible and heat this strongly. Test the escaping vapor with moist litmus paper (?). Write the equation for the thermal decomposition of the salt and name the residue. In quantitative analysis phosphorus and magnesium are usually determined by weighing them in this form.

(d) **Separation of Ca^{++} and Mg^{++} .** — Dissolve 0.5 g. of dolomite (formula ?) in dilute hydrochloric acid, heating to complete the reaction. Equation. A few particles will probably fail to dissolve. These will be silica, an impurity in the dolomite. Dilute the solution to about 20 cc. and filter out the insoluble particles. Dissolve a gram of solid ammonium chloride in the filtrate, make it distinctly alkaline with ammonium hydroxide, and add about 10 cc. of ammonium carbonate solution (?). Equation. Filter out the precipitate and add a solu-

tion of sodium phosphate to the filtrate (?). For what ion is this a test? Equation. Dissolve some of the precipitate on the filter paper by pouring acetic acid on it, catch this filtrate in a clean test tube and add ammonium oxalate solution (?). Equation.

EXERCISE 84

ZINC AND CADMIUM

Textbook: 631-636

✓ (a) **Basicity.** — Test solutions of a zinc salt and a cadmium salt with litmus paper (?). Explain. Name two other metals whose salts give a similar reaction.

✓ (b) **Hydroxides.** — Dilute 5 cc. of a solution containing zinc ions with an equal volume of water and add a solution of sodium hydroxide, drop by drop, until a considerable precipitate is obtained. Describe it and write the equation. Divide the precipitate into two portions, treat one with an excess of sodium hydroxide, and the other with dilute hydrochloric acid (?). Indicate the manner in which zinc hydroxide may ionize. (Refer to Exercise 68 and mention several other hydroxides which may act in this manner.)

Repeat the directions in the preceding paragraph, substituting cadmium for zinc. Compare the results with those obtained for zinc.

✓ (c) **Ammonia-Complex Ions.** — Add ammonium hydroxide, drop by drop at first and then in excess, to a solution of a zinc salt (?). Equations. Repeat this test using a cadmium salt solution and compare the results. Write the formulas for similar complexes formed by two other metals.

(d) **Sulfides.** — In this and subsequent experiments requiring the use of hydrogen sulfide gas, use an Erlenmeyer flask fitted with a one-hole stopper bearing a short piece of glass tubing to which about 15 cm. of small rubber tubing is attached. See Figure 28. The solution to be treated with H_2S is placed in this flask, the rubber tubing is connected to the hydrogen sulfide jet and the gas is turned on. The stopper should be loosened momentarily to allow some of the air to escape from the flask as it is displaced by H_2S . Hydrogen sulfide is quite poisonous and caution should be observed in its use. The method just described is designed to avoid the escape of the

gas into the atmosphere of the laboratory. Prepare a flask as described above and keep it for the purpose indicated.

Dilute 10 cc. of a zinc salt solution with 20 cc. of water and saturate this with hydrogen sulfide gas as directed above (?). Now add about 15 cc. of ammonium hydroxide and again treat with hydrogen sulfide (?). Explain. Shake the flask and pour 10-cc. portions of the suspension into each of two test tubes. In one test the solubility of the precipitate in acetic acid (?). To the other portion add dilute hydrochloric acid, a few drops at a time, shaking the tube after each addition, until the solution is just neutral and then until it just clears (?). Record the number of drops required to dissolve the precipitate after the solution has been neutralized. Now add some sodium acetate solution until the precipitate reappears. Explain how sodium acetate can increase the concentration of the sulfide ion in this solution and indicate the ionic equilibria which are involved.

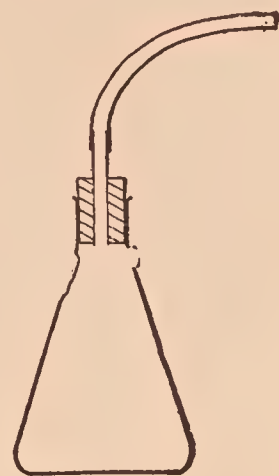


FIG. 28

Dilute 10 cc. of cadmium salt solution with 20 cc. of water and saturate the solution with hydrogen sulfide. Compare the appearance and amount of this precipitate with the zinc sulfide obtained under the same conditions. Shake up this precipitate and test the solubility of portions of it in acetic and dilute hydrochloric acids. The hydrochloric acid should be added a few drops at a time as suggested in the previous paragraph. Record the number of drops of acid required. Compare the solubilities of the two sulfides prepared.

(e) **Summary.** — Tabulate the products formed when zinc and cadmium ions react with sodium hydroxide, ammonium hydroxide, and hydrogen sulfide. Indicate likewise the physical and chemical properties of these products. How could you separate zinc hydroxide from cadmium hydroxide? How could you separate the sulfides of these two metals?

EXERCISE 85

MERCURY

Textbook: 637-642

✓ (a) **Mercurous Salts.** — Add some hydrochloric acid to a solution of mercurous nitrate (?). What other cation that you have studied would give a similar result? Allow the precipitate

to settle, pour off most of the liquid, and add ammonium hydroxide (?). Refer to your textbook for the composition of the product. Equation. This is a test for mercurous salts.

To small portions of mercurous nitrate solution add separately (1) sodium hydroxide solution; (2) potassium iodide solution; (3) ammonium hydroxide. Describe the results and write the equations.

Dilute 2 or 3 cc. of a mercurous salt solution and saturate it with hydrogen sulfide gas (?). In this and in certain other reactions mercurous salts react as if they consisted of a mercuric salt and mercury. The products in this reaction are, hence, mercuric sulfide and mercury. Equation.

✓(b) **Mercuric Salts.** — Add a few drops of hydrochloric acid to a solution of a mercuric salt. Compare the solubilities of the two chlorides of mercury. What are the common names of these two substances? Mention a use for each.

Add a solution of sodium hydroxide to a mercuric salt solution. What is the composition of the product? What other cations give a similar result? Is the precipitate soluble in excess sodium hydroxide? Compare the corresponding results for cadmium and zinc.

Add a solution of potassium iodide to a solution of a mercuric salt, first adding a small quantity and then an excess (?). What is finally formed?

To a solution of mercuric chloride add ammonium hydroxide until a definite result is obtained (?). Compare this with the corresponding reactions of zinc and cadmium. A solution of mercuric nitrate may be used for this test if the excess nitric acid in the solution is first carefully neutralized by adding the proper amount of sodium hydroxide solution before adding ammonium hydroxide. Perform this experiment and write the equations. Now add a solution of ammonium nitrate to the white precipitate (?). Is the reaction reversible?

Add a solution of stannous chloride to a solution of mercuric chloride, first a small amount and then an excess (?). Notice two distinct changes and write the equations. This test is used to identify mercuric salts.

Dilute 5 cc. of a mercuric salt solution and precipitate mercuric sulfide from this solution. Filter out the precipitate, place portions of it in porcelain dishes and test its solubility separately in hydrochloric and nitric acids and in aqua regia (?).

Heat these reagents to boiling when making the tests. When solution is accomplished evaporate it almost to dryness, take up the residue with water and test this solution by adding stannous chloride (?). Equations.

(c) **Preparation of Mercuric and Mercurous Nitrates.** — Place a small drop of mercury in a clean beaker and add 3 cc. of dilute nitric acid. Add one or two drops of concentrated nitric acid to start the chemical action. Allow this to proceed for several minutes, and then decant the solution into a test tube, retaining the remainder of the mercury in the beaker. Test the solution for mercurous and mercuric ions (?). Equations.

Add sufficient concentrated nitric acid to the remaining mercury to dissolve it completely and heat the beaker to accelerate the action. When the mercury has entirely disappeared test the solution for mercurous and mercuric ions (?). Equations. What would occur if metallic mercury were placed in the bottle containing mercuric nitrate solution?

EXERCISE 86

THE PREPARATION OF MERCURIC THIOCYANATE

Textbook: 642

(Pharaoh's Serpent Eggs)

Carefully counterpoise a small beaker on the balances, and in it weigh (to an accuracy of 0.1 gram) about 15 g. of either mercuric oxide or metallic mercury. To the oxide in the beaker add 15 cc. of concentrated nitric acid, or 20 cc. of concentrated nitric acid if metallic mercury is used, and warm gently until solution is complete. Evaporate the solution slowly over a low flame until a few crystals begin to appear on the surface of the liquid. What are they? Write the equation.

Wash this material into a 400-cc. beaker, using about 200 cc. of water altogether, and stir well. If a white suspension or cloudiness results, it indicates the formation of a basic salt and may be cleared up by adding a few drops of concentrated nitric acid and stirring. A faint cloudiness may be disregarded. Suggest a formula for this basic salt.

If a soluble thiocyanate is now added in an amount exactly equivalent to the Hg^{++} ions present, insoluble $\text{Hg}(\text{SCN})_2$ will be precipitated quantitatively. However, an excess of either

of the reagents increases the solubility of $\text{Hg}(\text{SCN})_2$ due to the formation of complex ions, and the yield of the precipitated product will be lowered proportionately. What is the usual effect of an excess of a common ion? In order to avoid an excess of either reagent it is necessary to use an indicator which shows when the end of the reaction is reached. For this purpose a ferric salt may be used since $\text{Fe}(\text{SCN})_3$ is a soluble substance and has an intense red color. If a solution of a thiocyanate is added to the solution of a mercuric salt containing a few drops of a ferric salt solution, the SCN^- ions combine with Hg^{++} ions as long as any of these are present, forming $\text{Hg}(\text{SCN})_2$ which appears as a precipitate toward the end of the reaction. When the Hg^{++} ions are used up and the SCN^- ions are added in excess, the latter combine with Fe^{+++} ions to form the red compound, thus indicating that the end of the principal reaction has been reached.

To the mercuric nitrate solution in the beaker add about 5 drops of ferric chloride solution and then add slowly and with constant stirring a solution of ammonium thiocyanate (or potassium thiocyanate) until a faint pink color persists in the solution. Equations.

Allow the precipitate to settle, decant the clear liquid and filter the remainder, using a large filter paper (about 18 cm.). Wash the precipitate in the filter paper until the washings no longer have a pink tint. Allow the precipitate to drain for some time, then open the filter paper and lay it flat in a warm place to dry or leave it in your desk until the next laboratory day.

When quite dry, the material should be carefully scraped from the filter paper into the original counterpoised beaker and weighed. Calculate the theoretical yield and your percentage yield.

Take several small portions of the material and test its solubility in a solution of mercuric nitrate and in a solution of the thiocyanate.

Mercuric thiocyanate, when made into pellets and ignited, burns to a coherent ash which assumes sinuous forms — hence called Pharaoh's serpents. The dried material should be moistened to a thick dough with dextrin solution and molded with the fingers into conical "eggs." When these are thoroughly dried they may be ignited under the hood on a piece of asbestos. (*Caution:* Do not breathe the fumes as they are poisonous.)

EXERCISE 87

ALUMINUM

Textbook: 652-657

(a) **Aluminum Hydroxide.** — Dilute 5 cc. of a solution of an aluminum salt with an equal volume of water and to this add sodium hydroxide solution slowly until a considerable amount of precipitate is obtained (?). Equation. Suspend the precipitate by shaking and divide it into three portions. To one portion add an excess of sodium hydroxide solution (?), to the second add dilute hydrochloric acid (?), and to the third add ammonium hydroxide (?). In which of these reagents is the precipitate readily soluble? Equations. Indicate the manner in which aluminum hydroxide may ionize. What term is applied to hydroxides which have this property? Compare zinc, cadmium and magnesium hydroxides with aluminum hydroxide, stating how you could distinguish these four substances from each other by means of reagents mentioned in this paragraph.

Test solutions of aluminum salts with litmus and conclude as to the strength of aluminum hydroxide as a base (?). What would occur if the aluminum salt of a weak acid were treated with water? Would the fact that aluminum hydroxide is practically insoluble have any bearing on the result? With these answers in mind, add some sodium carbonate solution to a solution of an aluminum salt (?). Equation. Add a solution of borax to a solution of an aluminum salt (?). Explain. Saturate 10 cc. of ammonium hydroxide with hydrogen-sulfide gas. Equation. Add this solution to the solution of an aluminum salt (?). Equation.

Precipitate a small quantity of aluminum hydroxide by means of ammonium hydroxide, filter out the precipitate and transfer some of it to a porcelain crucible. Heat this gently at first until it is dry and then to a red heat. Allow the crucible to cool and examine the residue. What is it? Equation. Is the reaction reversible? Test the solubility of the product in hydrochloric acid (?). What conclusion could you draw as to its melting point? For what is this substance used?

(b) **Baking Powder.** — Write the equation for the reaction of alum with sodium bicarbonate to liberate carbon dioxide.

It is suggested that you balance the equation by assuming the formation of aluminum bicarbonate which subsequently breaks down or is hydrolyzed. *aluminum hydroxide & carbon dioxide*

Calculate the weight of alum needed to react with 2 g. of sodium bicarbonate, weigh out these two substances, powder the alum, and mix them. Place the mixture in a small beaker and add 50 cc. of water (?). After effervescence has practically ceased identify the precipitate suspended in the solution (?).

(c) **Alum.** — Calculate the weight of potassium sulfate which will unite with 12 g. of aluminum sulfate crystals, $\text{Al}_2(\text{SO}_4)_3 \cdot 18\text{H}_2\text{O}$, to form an alum. Weigh out these quantities of the two salts, mix them in a beaker and add about 50 cc. of water. Heat the water to boiling to dissolve the salts and set it aside to crystallize. If a loop of thread is suspended in the solution by hanging the ends of the thread over opposite sides of the beaker, the crystals will form on the thread in a manner resembling rock candy.

After several days remove some of the crystals, dry them, and test them for aluminum, potassium, sulfate, and water of hydration. Write the equation for the formation of this alum. Mention two metals which would be substituted for aluminum in this formula.

What could be substituted for potassium? Write formulas for two other alums.

Leave some clear alum crystals exposed on a watch glass in your desk.

After several days examine them and discuss any change which you observe.

(d) **Alum as a Mordant.** — Dissolve about a gram of alum or aluminum sulfate in 15 cc. of water. Dilute 15 cc. of methyl orange solution with an equal volume of water. Obtain two pieces of white cotton cloth about 5 cm. $\times$ 10 cm. (Pieces of your towel will serve.) Dip one piece of cloth in half of the methyl orange solution and allow it to dry. Dip the second piece of cloth in the alum solution, squeeze out most of the solution, then dip it in the other half of the methyl orange solution and allow the cloth to dry. When the pieces of cloth have become entirely dry wash them thoroughly with water and compare the amounts of dye remaining in the two pieces. Explain.

EXERCISE 88

QUALITATIVE ANALYSIS OF BAKING POWDER

Unknown No. 3. — This material will be a sample of a commercial baking powder. One type of baking powder was studied in Exercise 71 (b). Other acidic substances commonly used in place of cream of tartar are calcium acid phosphate, potassium bisulphate, or sodium aluminum sulfate. Write the equations by which these substances would react with baking soda — refer to Exercise 87 (b). Starch is usually present to dilute the mixture and to keep it dry.

Mix about 2 g. of the baking powder with 30 cc. of water and identify the gas evolved (?). This indicates the presence of what anion? When effervescence has ceased, filter the suspension and use the residue for (a), reserving the filtrate for other tests.

(a) **Starch.** — Puncture the filter and wash the residue into a test tube with about 10 cc. of water. Boil this, allow it to cool, and test it for starch by adding a drop of iodine solution or a solution of potassium iodide to which chlorine water has been added.

(b) **Tartrate.** — To 5 cc. of the filtrate add 5 drops of concentrated sulfuric acid and evaporate to dryness. A charring of the residue and the characteristic odor of burnt sugar indicates a tartrate.

If the baking powder contains "soluble starch" this will interfere with the above test for a tartrate as it will give the same charred appearance. An additional test for a tartrate should be made as follows: Shake about a gram of the baking powder with 10 cc. of water until action ceases. Filter the solution, add 2 cc. of sodium hydroxide solution and mix well. Now add a solution of copper sulfate, drop by drop, shaking the tube, until about 8 drops have been added. If a light blue precipitate of copper hydroxide does not appear the presence of a tartrate is indicated. What use was made of this reaction in Exercise 76 (c)?

(c) **Sulfate.** — To 5 cc. of the filtrate add barium chloride solution. A precipitate insoluble in dilute hydrochloric acid indicates a sulfate. Carbonates and phosphates give precipitates which are soluble in acids.

(d) **Phosphate.** — To 5 cc. of ammonium molybdate solution add about 1 cc. of the filtrate and acidify the mixture with nitric acid. Warm the mixture gently and allow it to stand. The formation of a yellow precipitate indicates a phosphate. *no precip*

(e) **Aluminum.** — Refer to Exercise 87 (b) and decide whether most of the aluminum would be retained by the filter or would pass through in solution (?). Test some of the filtrate for aluminum (?). Devise and perform a test for aluminum using some of the original sample.

(f) **Calcium.** — Shake a small quantity of the baking powder with dilute hydrochloric acid. Filter and neutralize the filtrate with ammonium hydroxide. Now make the solution slightly acid with acetic acid, heat to boiling and add a solution of ammonium oxalate. What result do you expect if calcium is present? *white ppt*

(g) **Sodium and Potassium.** — Moisten a clean platinum wire with hydrochloric acid, dip the wire in the sample of baking powder and test it in the flame (?). View the flame through cobalt glass to test for potassium. If these metals are indicated by the flame tests they should be confirmed by tests studied in Exercise 73. If aluminum and calcium are present these should be removed by precipitation with ammonium carbonate. The filtrate from this precipitate should be evaporated to dryness and the ammonium salts removed (?); the residue from this treatment, dissolved in water, may be tested for sodium and potassium.)

From your results indicate the substances present in the sample of baking powder. Report this conclusion as the analysis of your unknown.

EXERCISE 89

TIN

Sample of baking powder to contain
Exercise 89
Textbook: 666-669

Textbook: 666-669

(a) **Stannic Compounds.** — Dilute 5 cc. of stannic chloride with an equal volume of water and treat the solution with hydrogen sulfide (?). Equation. Filter out the precipitate, transfer most of it to a porcelain dish and add 3 or 4 cc. of yellow ammonium sulfide, $(\text{NH}_4)_2\text{S}_x$. Warm the dish gently (do not boil) and stir the mixture until the precipitate dissolves.

What is formed? Equation. Filter this solution into a test tube and acidify it with dilute hydrochloric acid (?). Compare this result with that obtained when some yellow ammonium sulfide is acidified. Equations. This experiment is used as a test for stannic compounds.

(b) **Stannous Compounds.** — Repeat the above experiment throughout using stannous chloride in place of stannic chloride. Compare the result at each point with the corresponding result for the stannic compound, and write all the equations. These reactions are used to identify stannous compounds.

Add a few drops of mercuric chloride solution to 2 cc. of stannous chloride solution. Note two distinct changes, adding more stannous chloride if necessary. Equations. State how this experiment may be used as a test for one type of mercury compound and conversely for one type of tin compound.

Add a few drops of potassium dichromate solution to 5 cc. of stannous chloride solution (?). Write the equation taking account of the presence of excess HCl in the stannous chloride solution. Repeat this test using potassium permanganate solution instead of the dichromate (?). Equation. What seems to be the chief chemical property of stannous chloride?

To 5 cc. of stannous chloride solution add a solution of sodium hydroxide, slowly at first and then in excess (?). Write two equations to represent the changes which you observe. Name the final product. Add portions of this solution to: (1) a solution of bismuth chloride; (2) the white precipitate, BiOCl , made by diluting bismuth chloride solution with water; (3) bismuth hydroxide, made by adding ammonium hydroxide to a solution of bismuth chloride. The black product is metallic bismuth. Write the equations.

(c) **Action of Tin with Acids.** — Treat a small piece of tin with 5 cc. of concentrated hydrochloric acid in a small beaker. Warm the beaker gently to accelerate the action, allow this to proceed for some time, and finally evaporate the liquid almost to dryness. Allow the beaker to cool. Is there any evidence of a crystalline product? Add 20 cc. of water to the beaker to dissolve the product, discard any tin which remains undissolved and test the solution to determine whether stannous or stannic chloride was produced (?).

Add a few drops of concentrated nitric acid to a small piece of tin (?). Describe the product. What is it? How could you

distinguish tin from zinc and from aluminum by means of nitric acid? If you are in doubt try the experiment.

To a very small piece of tin add 3 cc. of concentrated hydrochloric acid and 1 cc. of concentrated nitric acid. What is this mixture of acids called? Allow the preparation to stand until all the tin has been dissolved, dilute the solution with 20 cc. of water and determine whether a stannous or a stannic compound has been formed (?). Write the equation for the solution of tin in this mixture of acids.

(d) **Preparation of Stannic Chloride.** — Place 2 g. of tin in a dry test tube, immerse this for most of its length in a bottle of cold water, and pass chlorine into the test tube until the tin has been converted to stannic chloride. The chlorine must be dried by passing it through a drying bulb filled with granular calcium chloride. Design your apparatus and a suitable method for generating the chlorine and obtain the approval of your instructor before proceeding with the experiment.

Describe the stannic chloride you obtain. Determine its boiling point as follows: Support the test tube containing the stannic chloride with a clamp attached to an iron stand. Wipe the tube dry on the outside. Obtain a cork stopper which is considerably larger than the mouth of the test tube, bore a hole in the cork and insert a thermometer. By resting the cork on the top of the test tube, suspend the thermometer in the tube so that the bulb of the thermometer is 2 cm. above the surface of the liquid. Boil the liquid, using a very low flame, so that the vapors do not rise much above the middle of the test tube but condense on the sides and run down. When a constant temperature is obtained, record it as the boiling point of the liquid.

When the stannic chloride is cool, pour a portion of it (with care) into some water (?). Suggest a method for recovering tin from "tin plate," that is, iron plated with tin.

EXERCISE 90

LEAD

Textbook: 673-680

(a) **Test for Lead.** — Dilute 5 cc. of lead nitrate with an equal quantity of water and add hydrochloric acid until precipitation is complete (?). Filter out the precipitate and dis-

solve it by pouring successive small portions of boiling water on the precipitate in the filter, catching the solution in a beaker. To small portions of this hot solution add separately: (1) dilute sulfuric acid (?); (2) a solution of potassium dichromate (?); (3) hydrogen sulfide solution or gas (?). Write the equations and describe the products by which you identify lead.

(b) **Lead Hydroxide.** — Add a solution of sodium hydroxide, drop by drop, to 5 cc. of lead nitrate solution until a considerable amount of precipitate has been formed (?). Suspend the precipitate by shaking, divide it into three portions and test its solubility in: (1) excess sodium hydroxide solution; (2) ammonium hydroxide; (3) acetic acid. Write equations to illustrate the results obtained. How does lead hydroxide differ in behavior from zinc hydroxide?

(c) **Oxides of Lead.** — Obtain small samples of lead monoxide, lead dioxide, and red lead. Give the formulas, common names, and a brief description for these three compounds. Test the solubility of each oxide in dilute nitric acid, warming the acid if necessary. Does this experiment give any indication of the probable constitution of red lead? Explain.

Treat small quantities of each of the three oxides with concentrated hydrochloric acid (?). Equations. Which of these oxides oxidize HCl?

Add bromine water to 5 cc. of sodium hydroxide solution until a yellow color is perceptible in the solution. Add this solution to 5 cc. of a solution of lead acetate (?). Transfer this mixture to a small beaker and boil it for several minutes (?). Identify the product by means of appropriate tests and write the equations resulting in its formation.

(d) **Separation of Pb^{++} , Hg^+ , and Ag^+ .** — Mix 2 cc. of lead nitrate solution, 2 cc. of mercurous nitrate solution, and 2 cc. of silver nitrate solution in a test tube or small beaker and add dilute hydrochloric acid until no further precipitation occurs. Filter out the precipitate, wash it with 10 cc. of cold water, and treat it on the filter paper with small portions of boiling water. Catch the solution which runs through and test portions of this for lead by at least two methods. Continue the treatment with hot water until the washings which run through do not give a test for lead.

To the residue on the filter add ammonium hydroxide and catch the solution which runs through. Acidify the filtrate with dilute nitric acid and identify the precipitate (?). Ex-

amine the residue on the filter. What is it? Recall Exercise 85 (a). Write the equations for the reactions involved in the separation and identification of these three ions.

(e) **Unknown No. 4.** — Test this unknown solution for lead, silver, and mercurous ions by the procedure outlined in (d).

EXERCISE 91

CHROMIUM

Textbook: 688-692

(a) **Chromic Hydroxide.** — Precipitate some chromic hydroxide by the addition of either sodium hydroxide solution or ammonium hydroxide to a solution of a chromic salt. Test the solubility of this precipitate in: (1) excess ammonium hydroxide; (2) a dilute acid; (3) excess sodium hydroxide. Write equations. Boil the clear solution in (3) until a change occurs (?). Transfer about 2 cc. of this suspension to a small beaker and add about a gram of sodium peroxide, a very little at a time, with stirring. Add about 10 cc. of water and boil to decompose the excess sodium peroxide. A yellow color denotes the presence of sodium chromate. Write the equation for its formation. To a portion of the solution add a solution of barium chloride (?). Equation. Save a portion of the solution for (b).

(b) **Chromates and Dichromates.** — Acidify the solution reserved from (a) with dilute nitric acid and note the change in color which occurs (?). Equation. Add sodium hydroxide solution to a solution of potassium dichromate until a change in color is noted (?). Equation.

Add 2 drops of potassium dichromate solution to 5 cc. of water, acidify the solution with 2 drops of dilute sulfuric acid, add about 2 cc. of ether and 1 cc. of hydrogen peroxide solution. Shake the tube and note the color in the ether layer (?). This may be used as a test for chromates and dichromates, or conversely for hydrogen peroxide. It is not a test for chromic salts.

To small portions of potassium dichromate solution add separately: (1) a solution of barium chloride; (2) a solution of a lead salt; (3) a solution of silver nitrate. The precipitates are chromates and may be used as identifying tests — recall Exercises 81 (b) and 90 (a). Write the equations for the formation of the precipitates. Divide the precipitate in (1) into two

portions and test the solubility of barium chromate in acetic acid and in dilute hydrochloric acid (?).

What is the action of a reducing agent on a dichromate? Illustrate your answer by means of an equation.

Outline a test for a chromic salt.

(c) **Chromic Anhydride.** — Dissolve 5 g. of sodium dichromate in 10 cc. of water, warming it to hasten the solution. This salt is much more soluble than potassium dichromate. Cool the solution and add with care about 15 cc. of concentrated sulfuric acid. This mixture is used in laboratories as a cleaning solution for glassware. Allow the solution to cool slowly and to stand for some time. Filter out the crystals using a small plug of glass wool in the stem of the funnel. Why may a filter paper not be used?

Examine and describe the crystals. Dissolve a few of them in water (?). Add lead nitrate solution to the solution of the crystals (?). Pour some sodium hydroxide solution over the remainder of the crystals in the funnel and catch the solution which filters through. Judging from its color what compound is present? Acidify this solution with nitric acid (?).

(d) **Preparation of Ammonium Dichromate.** — Dissolve 10 g. of chromic anhydride in 20 cc. of water and add the calculated amount of concentrated ammonium hydroxide (sp. gr. = 0.9; 28 per cent NH_3). Do not allow the solution to become too hot when mixing because much of the ammonia will escape if this occurs. Pour the solution into a beaker and allow it to crystallize.

When crystals have formed, filter them out and dry them with filter paper. Place the dry crystals in a conical pile on an asbestos mat and ignite them by means of a piece of fuse paper (?). Write the equations for the formation and decomposition of ammonium dichromate.

EXERCISE 92

PREPARATION OF CHROME ALUM

Textbook: 690

Construct the equation for the reduction of potassium dichromate by hydrogen sulfide in the presence of sulfuric acid. Weigh out 15 g. of potassium dichromate, place this in an

Erlenmeyer flask and add 100 cc. of water. Calculate the volume of concentrated sulfuric acid (sp. gr. = 1.84; 95 per cent H_2SO_4) required for this weight of the dichromate according to the equation you have written. Add this volume of acid slowly to the water in the flask and heat the mixture to boiling to dissolve the potassium dichromate.

Pass hydrogen sulfide gas into this hot solution in the manner described in Exercise 84 (*d*), shaking the flask vigorously at intervals. This treatment should be continued until the solution in the flask is a dark green and will require 30 minutes or longer.

When the reduction of the dichromate is complete, pour the solution into a beaker and boil it for about 5 minutes to coagulate the sulfur and expel the excess hydrogen sulfide. Filter this solution while hot and set the filtrate aside to cool. Describe the residue on the filter.

Crystallization of the alum will be facilitated by seeding the solution with crystals which may serve as nuclei for growth. Since all the alums have the same crystalline form (are isomorphous) crystals of any alum may be used as the seed. When the filtered solution of chrome alum is quite cool, drop in a piece of ordinary alum about the size of a grain of corn and place the solution in your desk to crystallize.

After several days pour off the mother liquor into another beaker and examine the crystals. Compare the color of the crystals by transmitted light with the color of the solution (?). Pick out the crystal which grew on the seed, break it through the center and describe its appearance. Dry a crystal with filter paper and leave it exposed to the atmosphere for some time. Dissolve some of the chrome alum crystals in water (color?) and test portions of the solution for the chromic ion, potassium ion and sulfate ion.

Mixed Crystals. — Prepare 10 cc. of a saturated solution of common alum at about 40° . Using 10 cc. of the mother liquor from the chrome alum and a few of the chrome alum crystals, prepare a similarly saturated solution. Mix these two solutions, cool the mixture to room temperature and, if no crystals are visible in the solution, seed it with a very small crystal of any alum. After sufficient growth has taken place examine and describe the crystal comparing it with chrome alum and with ordinary alum. What is the chemical composition of this crystal? How could you prove your answer?

EXERCISE 93

MANGANESE

(a) **Manganous Hydroxide.** — Dilute 5 cc. of a manganous salt solution with 15 cc. of water, boil this solution to expel dissolved oxygen, and divide the solution into four portions. To a portion of the solution add ammonium hydroxide (?). Is the precipitate soluble in an excess of this reagent?

To a portion of the solution add an equal volume of ammonium chloride solution and then add ammonium hydroxide (?). Recall Exercise 72 (a) and explain the result of this test.

To the third portion of the manganous salt solution add a solution of sodium hydroxide (?). Test the solubility of this precipitate in dilute nitric acid (?).

Add sodium hydroxide to the fourth portion of the solution, then add 5 cc. of water and shake the tube to saturate the solution with air. Note and explain the gradual change in the color of the precipitate. Test the solubility of this precipitate in dilute nitric acid (?). Now add some hydrogen peroxide solution and shake the mixture (?).

Precipitate another small portion of manganous hydroxide and treat the precipitate in the alkaline solution with a solution of hydrogen peroxide (?). Explain the effect of hydrogen peroxide in the two instances, noting its action in acid solution and in alkaline solution. Write the equations.

(b) **Manganates and Permanganates.** — Make a mixture of about 0.5 g. of manganese dioxide with 2 g. of potassium carbonate and 2 g. of potassium nitrate. Place this mixture in an iron crucible and heat it until all of the material fuses together. A green color in the melt indicates the presence of a manganate.

Allow the crucible to cool and extract the mass with cold water, noting the color of the solution. Boil a portion of this solution and, if this does not change its color, pass carbon dioxide gas into it (?). Write the equation for the formation of potassium manganate assuming that the other products are carbon dioxide and potassium nitrite. Write an equation to explain the change in color of a manganate solution in presence of carbon dioxide.

Add 5 cc. of concentrated nitric acid to a few milligrams of

freshly prepared manganese dioxide and note that the oxide is not soluble. Now add a small amount of lead dioxide, boil the mixture for several minutes, add 10 cc. of water and filter the mixture. Note and explain the color of the filtrate. The products of the reaction are the acid which gives the colored ion you observed, lead nitrate, and water. Write the equation.

EXERCISE 94

IRON — FERRIC AND FERROUS COMPOUNDS

Textbook: 722-727

(a) **Tests for Ferric Iron.** — Dilute 2 cc. of a solution of ferric chloride with 10 cc. of water, divide the resulting solution into 4 portions and add the following reagents to separate portions: (1) sodium hydroxide; (2) potassium ferrocyanide; (3) potassium ferricyanide; (4) a soluble thiocyanate. Write equations.

(b) **Tests for Ferrous Iron.** — Repeat the tests in (a) using a diluted solution of a ferrous salt. The thiocyanate test gives no color with ferrous compounds. If a red color is obtained in this test what is your conclusion as to the action of air upon a solution of a ferrous salt? Pour 2 or 3 cc. of ferrous sulfate solution into a test tube, add 1 cc. of dilute sulfuric acid and a few iron filings. Boil this mixture gently for some time, dilute the product with 10 cc. of water, allow the filings to settle to the bottom and try the thiocyanate test on this solution (?). Draw a conclusion from this test, whatever the result (?).

Tabulate the results of (a) and (b) indicating the distinguishing colors and the important tests for the two kinds of iron ions.

(c) **Oxidation — Reduction.** — Boil 5 cc. of ferrous sulfate solution with iron filings and dilute sulfuric acid. Decant the solution into another test tube, add concentrated nitric acid, drop by drop, and boil the solution until further addition of nitric acid does not develop a brown color. Note the color of the solution (?). Equation. Test the solution for ferrous and ferric iron (?).

Prepare a small amount of ferrous hydroxide, filter it and leave the precipitate exposed on the filter paper for 10 minutes. Note any change in color (?). Dissolve this precipitate by pouring dilute hydrochloric acid on the filter and test the solution for iron of each valence (?).

Dilute 2 cc. of a ferric salt solution with 10 cc. of water, heat the solution and treat it with hydrogen sulfide gas. Note a

^{white} change in color and the formation of a precipitate (?). Equation. Determine by test the resulting valence of the iron. ^{add this cy}

(d) **Ferrous Sulfide.** — Precipitate some ferrous sulfide from a solution of ferrous ^{sulfate} sulfate (?). Equation. Describe the precipitate and test its solubility in dilute acid. ^{sol} What use is made of ferrous sulfide in chemical laboratories? Given a solution containing AgNO_3 , $\text{Sn}(\text{NO}_3)_4$ and $\text{Fe}(\text{NO}_3)_2$, outline a procedure for the separation and identification of each cation. ^{add this cy}

(e) **Hydrolysis of Ferric Chloride.** — Test a solution of ferric chloride with litmus paper (?). Construct an ionic equilibrium to explain the result. ^{red}

Pour a few drops of a ferric chloride solution into a small quantity of boiling water and note the change in color. Refer to Exercise 23 and explain.

EXERCISE 95

CORROSION OF IRON

Textbook: 720-722

Attach a small piece of zinc to the head of a clean iron nail. Be sure to obtain good contact between the two metals. Similarly attach a piece of tin to another nail. Place these in separate test tubes and in a third tube place an ordinary iron nail.

Prepare some "ferroxyl reagent" as follows: Boil about 0.5 g. of agar in 50 cc. of water until the agar has been disintegrated and taken into colloidal solution. Now add 2 drops of phenolphthalein solution and a few drops of potassium ferricyanide—enough to give a slight yellow tint to the solution. Pour enough of this solution into the test tubes to cover the nails, stopper the tubes, and set them where they will not be disturbed.

Examine the tubes at intervals for several days. The purpose of the agar was to form a jelly which would hold the materials in place. Where the iron goes into solution (rusts) a blue precipitate of ferrous ferricyanide, $\text{Fe}_3[\text{Fe}(\text{CN})_6]_2$, will be formed. Compare the nails as to the amount and location of this action. Points in the solution at which hydrogen has been deposited will have an excess of OH^- ions and will appear pink due to the phenolphthalein.

From this experiment and from the principles noted in Exercise 78, draw conclusions as to the efficacy of zinc and tin in preventing the rusting of iron.

EXERCISE 96

PREPARATION OF DOUBLE SALTS

A double salt may be formed by mixing solutions of the simple salts composing it in the proportions required by the formula of the double salt. Recall the double salts prepared in this manner in Exercises 77 (c), 87 (c) and 92. When crystals of the double salt are dissolved all the ions are given which would be formed by the component simple salts. This is not true of a complex salt such as $\text{K}_4\text{Fe}(\text{CN})_6$. What ions does potassium ferrocyanide give? How should its formula be written if it were a double salt?

(a) $\text{FeSO}_4 \cdot (\text{NH}_4)_2\text{SO}_4 \cdot 6\text{H}_2\text{O}$. — Calculate the weight of iron which will dissolve in 50 cc. of dilute sulfuric acid (6N). Use about a gram in excess of this weight of clean iron wire, place it in 50 cc. of dilute sulfuric acid and allow the reaction to proceed until no more hydrogen is evolved. Remove any iron which has not dissolved, filtering the solution if necessary.

Neutralize another 50 cc. of dilute sulfuric acid with a concentrated solution of ammonia and mix this solution with the solution of ferrous sulfate you have just prepared. Heat the mixture to boiling, evaporate it to about 75 cc., and set it aside to crystallize. Calculate the weight of the double salt crystals which could be formed from the amounts of reagents used.

When the solution has cooled and a sufficient crop of crystals has appeared, filter out the crystals and reserve the filtrate for (b). Dry the crystals by exposing them to air on filter papers and weigh them. Measure the volume of the filtrate and calculate the weight of the double salt which it must still contain at 20° if the solubility of the salt is 22 parts per 100 parts of water at this temperature. Why will this calculation be slightly inaccurate even if the temperature of the filtrate is exactly 20° ? What weight of the crystals should you have obtained theoretically? What is your percentage yield?

Are the crystals of this double salt efflorescent? Is the salt oxidized by air to a ferric compound? Test the crystals for ferrous and ferric iron, for the ammonium and sulfate ions, and for water of hydration. Write equations for all the chemical reactions which you have employed in this experiment?

(b) $\text{Fe}_2(\text{SO}_4)_3 \cdot (\text{NH}_4)_2\text{SO}_4 \cdot 24\text{H}_2\text{O}$. — To the filtrate from (a) add, with care, 5 cc. of concentrated sulfuric acid. Heat the solution to boiling and add concentrated nitric acid, a little at a time, with stirring, until a drop of the solution in 2 cc. of water fails to give a test for the ferrous ion.

Evaporate the solution to about one-half its volume, place a string across the beaker so that it hangs in the solution, and set the solution in your desk to crystallize.

At the next laboratory period examine and describe the crystals. To what class of substances does this double salt belong? Write the formulas and a brief comparison of the two other substances of this type which you have prepared.

EXERCISE 97

COBALT

Textbook: 728-730

(a) **Cobaltous Hydroxide.** — Add a solution of sodium hydroxide to a solution of a cobaltous salt. Describe the precipitate which is formed. Is it soluble in excess sodium hydroxide? Repeat the experiment with ammonium hydroxide in place of sodium hydroxide (?). Why does the precipitate dissolve in excess ammonium hydroxide? Note the color of the solution. Equation.

(b) **Cobaltous Sulfide.** — Treat 10 cc. of a solution of a cobalt salt with hydrogen sulfide (?). Now add an excess of ammonium hydroxide to the solution and again treat it with hydrogen sulfide (?). Equation. To what group of sulfides does cobalt sulfide belong? Refer to Exercise 49 (b). Filter out the precipitate, remove a small portion and test its solubility in dilute hydrochloric acid (1.2 normal) prepared by diluting 1 part of the concentrated acid with 9 parts of water (?).

Prepare a borax bead as in Exercise 59 (b), take up a particle of the cobalt sulfide on this bead and heat it in the oxidizing flame of the Bunsen burner. The cobalt sulfide will be oxidized in the flame to cobalt oxide which will react with borax to produce a mixture of the metaborates of cobalt and sodium. Note the color and write the equation.

Transfer the remainder of the cobalt sulfide to a porcelain

evaporating dish and dissolve it by boiling it with the minimum quantity of a mixture of 3 parts dilute hydrochloric acid with 1 part dilute nitric acid (dilute aqua regia). Equation. Evaporate this solution almost to dryness, take up the residue in water and filter it. This solution of cobaltous chloride should be reserved for use in (c).

(c) **Sympathetic Ink.** — Dip a splint of wood or a glass rod in a dilute solution of cobalt chloride and write upon a piece of white paper. Permit this to dry and note whether or not the writing is visible. Warm the piece of paper over a flame being careful to avoid scorching the paper (?). Now breathe upon the paper (?). Explain the changes observed. To 1 cc. of a solution of cobalt nitrate add concentrated hydrochloric acid until a change in color is observed. Suggest the cause of this change.

Many other sympathetic or secret inks can be devised. For example, writing with a solution of a lead salt can be developed by a blotter or filter paper moistened with a solution of hydrogen sulfide; writing with potassium ferrocyanide can be developed by means of a solution of a ferric salt. Suggest other possible combinations.

EXERCISE 98

NICKEL

Textbook: 730-732

(a) **Nickelous Hydroxide.** — To a solution of a nickelous salt add a solution of sodium hydroxide (?). Is the precipitate soluble in excess? Repeat the experiment substituting ammonium hydroxide as the precipitant (?). What is formed when excess ammonium hydroxide is added? The color resembles that of what other ammonia complex? Write the formulas for all the metal-ammonia ions which you have encountered in this course and note any characteristic colors.

(b) **Nickelous Sulfide.** — Treat a solution of a nickel salt with hydrogen sulfide (?). Now make the solution alkaline with ammonium hydroxide and again treat with hydrogen sulfide (?). Test the solubility of the precipitate in 1.2 normal hydrochloric acid and in dilute aqua regia (?). Compare the results with those obtained in Exercise 97 (b). Make a borax-bead test with nickel sulfide and compare it with the bead test for cobalt.

(c) **Separation of Nickel and Cobalt.** — Test separately small portions of solutions of nickel and cobalt salts with a solution of dimethyl-glyoxime. Describe the results and suggest a method for the separation and identification of nickel and cobalt.

EXERCISE 99

UNKNOWN NO. 5 — "GROUP UNKNOWN"

The qualitative Groups I to IV are to be reported if present. No analysis of the precipitate is required.

GROUP	PROCEDURE	PRECIPITATE MAY CONTAIN
I	^{10 cc.} ~~Dilute 4 cc. of the unknown solution with 10 cc. of water and~~ add 4 cc. of dilute HCl. Filter; test small portion of filtrate with HCl to determine if precipitation was complete.	AgCl, ~~(H₂O₃)~~ <i>test - clau</i> Hg ₂ Cl ₂ , <i>with H₂O</i> ~~PbCl₂~~ <i>Ag - b</i> <i>8</i> <i>125</i>
II	Dilute filtrate to 100 cc. and saturate with H ₂ S. Heat nearly to boiling and filter.	~~PbS, HgS, As₂S₃,~~ ~~Sb₂S₃, Bi₂S₃,~~ ~~SnS, SnS₂,~~ CuS, CdS.
III	To the filtrate from II add NH ₄ OH to alkalinity (litmus test) and saturate with H ₂ S. Filter.	Al(OH) ₃ , ~~Cr(OH)₃,~~ FeS, ~~MnS,~~ ZnS, ~~CoS, NiS~~
IV	Acidify the filtrate from III with dil. HCl and evaporate the solution to 25 cc. in an open beaker. Filter if a precipitate (probably S) appears. Make the solution alkaline with NH ₄ OH, heat to boiling, and add (NH ₄) ₂ CO ₃ solution. Allow to stand.	CaCO ₃ , SrCO ₃ , <i>one</i> BaCO ₃ .
V	The filtrate from IV would contain Na, K, NH ₄ , and Mg as ions. There is no group reagent for Group V. It is not to be reported in this unknown.	

EXERCISE 100

UNKNOWN NO. 6 — "GENERAL UNKNOWN"

Not more than three cations (in all groups)

Procedure: The group separations are to be carried out as directed for Unknown No. 5. The group precipitates and the final filtrate (containing Group V) are to be analyzed as follows:

Test for NH_4 salts using a portion of the original unknown.

Group I

Proceed as with Unknown No. 4 — Exercise 90' (*d, e*).

Group II — Separation of Tin and Copper Sub-groups

(1) Wash the precipitate with water, transfer to a dish or beaker, add 10 cc. of $(\text{NH}_4)_2\text{S}_x$ solution, stir and warm (do not boil) for 10 minutes. Add 10 cc. of water and filter. Residue is Copper Group, filtrate contains Tin Group.

Filtrate — Analysis of Tin Group

(2) Dilute and acidify with dil. HCl ; boil, filter and wash precipitate. Discard filtrate, transfer precipitate to dish and heat it 10 minutes with 10 cc. of conc. HCl . Dilute, filter and wash the precipitate.

Precipitate — As_2S_5 and S

(3) Add 10 cc. of dil. HNO_3 and warm. Dilute, filter, and evaporate the filtrate to 5 cc. Add magnesia mixture and conc. NH_4OH until strongly alkaline. White precipitate on standing is $\text{MgNH}_4\text{AsO}_4$.

Filtrate — SbCl_3 and SnCl_4

(4) Dilute to exactly 50 cc., heat to boiling and saturate hot with H_2S for 5 minutes. Filter and wash the precipitate.

Precipitate — Sb_2S_3 (orange)

(5) Dissolve in conc. HCl , add aluminum wire. Black precipitate insoluble in HCl is Sb.

Filtrate — SnCl_4

(6) Evaporate to 30 cc., add one piece Al wire and heat until all action ceases. Filter into a solution of HgCl_2 . White or gray ppt. shows Sn.

Residue (from 1) — Analysis of Copper Group

(7) Add 5 cc. of dil. HNO_3 to 10 cc. of water, add this mixture to the residue in a beaker and boil for several minutes. Filter, wash ppt.

Residue (from 7) — HgS (black) and S

(8) Boil with 3 or 4 cc. of aqua regia until NO_2 is no longer evolved, dilute with 10 cc. of water and filter. Add SnCl_2 to filtrate (?).

Filtrate — Nitrates of Pb, Bi, Cu, Cd

(9) Add 3 cc. of conc. H_2SO_4 and evaporate in a beaker until dense white fumes of SO_3 appear. Allow to cool and pour into 15 cc. of water — rinse out first beaker with this solution. Stir and allow to stand; filter.

Precipitate — PbSO_4 (white)

(10) Dissolve ppt. in hot ammonium acetate solution and add $\text{K}_2\text{Cr}_2\text{O}_7$. A yellow ppt. insoluble in $\text{HC}_2\text{H}_3\text{O}_2$ is PbCrO_4 .

Filtrates — Sulfates of Bi, Cu, Cd

(11) Add NH_4OH , stirring well, until the solution is alkaline — then add 2 cc. NH_4OH in excess. Filter. Precipitate is $\text{Bi}(\text{OH})_3$. Add Na_2SnO_2 — black residue is Bi.

Filtrate — Cu and Cd as ammonia-complex sulfates

(12) If colorless, copper is absent — proceed with (14). If blue, save most of solution for (13) — acidify a small portion with H_2SO_4 and add 2 drops of $\text{K}_4\text{Fe}(\text{CN})_6$. Brown precipitate is $\text{Cu}_2\text{Fe}(\text{CN})_6$.

(13) Add an excess of dil. H_2SO_4 and about 5 g. of iron filings, cover with watch glass and boil several minutes. Filter and treat filtrate by (14).

Filtrate — Cd or $\text{Cd}(\text{NH}_3)_4$ as sulfate

(14) Acidify if necessary and saturate with H_2S . Yellow precipitate is CdS . Explain how Cu but not Cd was removed in (13) and why Fe ions do not interfere with the test for Cd in (14).

Group III — Separation of Fe and Al Sub-groups

Precipitate — $\text{Al}(\text{OH})_3$, $\text{Cr}(\text{OH})_3$, ZnS , MnS , FeS , CoS , NiS

(15) Transfer to beaker and add dil. HCl . If black residue (NiS , CoS) remains add a few drops of conc. HNO_3 and boil. Add 10 cc. water and filter out S. Evaporate filtrate to 10 cc. Add NaOH in excess and then Na_2O_2 slowly with stirring. Boil, dilute, filter and wash residue. Analyze filtrate by (16, 17); residue by (18–20).

Aluminum Group

Filtrate — NaAlO_2 , Na_2CrO_4 , Na_2ZnO_2

(16) Add HNO_3 to acidify, then NH_4OH to alkalinity, stirring well. Heat to boiling and filter. White precipitate is $\text{Al}(\text{OH})_3$.

Filtrate — Na_2CrO_4 (yellow), $\text{Zn}(\text{NH}_3)_4$ ions

(17) Add $\text{HC}_2\text{H}_3\text{O}_2$ to acidity — $\text{Na}_2\text{Cr}_2\text{O}_7$ and $\text{Zn}(\text{C}_2\text{H}_3\text{O}_2)_2$ are formed. Yellow or orange color indicates Cr — confirm by test in Exercise 91 (b). To the larger portion of the solution add 2 drops $\text{K}_4\text{Fe}(\text{CN})_6$ and warm. A white precipitate is $\text{Zn}_2\text{Fe}(\text{CN})_6$.

Iron Group

Residue (from 15) — MnO_2 , $\text{Fe}(\text{OH})_3$, $\text{Ni}(\text{OH})_2$, $\text{Co}(\text{OH})_3$

(18) Transfer to a porcelain dish, add 15 cc. conc. HNO_3 and heat to boiling. If a black residue remains add NaNO_2 solution until residue dissolves. Boil until NO_2 is no longer evolved. Add gradually, with stirring, 1 g. solid KClO_3 and boil for 3 minutes. A brown or black residue is MnO_2 . Filter through a plug of asbestos wool and wash. Transfer precipitate to a beaker, add 1 g. PbO_2 and 10 cc. dil HNO_3 . Boil and allow solids to settle. Pink color indicates permanganate.

Filtrate — Nitrates of Fe, Co, Ni

(19) Add NH_4OH in excess, filter. Precipitate is $\text{Fe}(\text{OH})_3$. Dissolve it in dil. HCl , add NH_4SCN . Red solution is $\text{Fe}(\text{SCN})_3$.

Filtrate — Ni, Co as ammonia-complex ions

(20) Acidify with $\text{HC}_2\text{H}_3\text{O}_2$, divide into two portions:

- (A) Add dimethyl glyoxime. Red precipitate indicates Ni; brown solution Co.
 - (B) Evaporate second portion to 2 cc., and add 2 cc. NH_4SCN and 2 cc. amyl alcohol-ether mixture and shake. Blue-green layer indicates Co.
-

Group IV — CaCO_3 , SrCO_3 , BaCO_3

(21) Dissolve the precipitate by repeatedly pouring 10 cc. of hot $\text{HC}_2\text{H}_3\text{O}_2$ through the filter and analyze the solution as in Exercise 81 (*c, d*).

Group V — Mg, K, Na, NH_4 Salts**Filtrate from Group IV**

(22) Add 5 cc. conc. HNO_3 and evaporate to dryness in a small beaker. Moisten residue with conc. HNO_3 and ignite to decompose NH_4NO_3 . Cool, add 10 cc. water, a few drops HCl and filter if not clear. Test one-third of filtrate by (23) and two-thirds by (24).

(23) Add conc. NH_4OH through a filter until solution is alkaline. If a precipitate forms dissolve it by adding 3 cc. NH_4Cl solution through the filter. Then add 2 cc. Na_2HPO_4 through the filter, shake the mixture vigorously and allow to stand. A white ppt. is MgNH_4PO_4 .

(24) Make flame tests and precipitation tests for Na and K as in Exercise 73.

If any difficulties are met consult a manual of Qualitative Analysis or an instructor.

Additional unknowns consisting of alloys, everyday substances, etc., may be given for further practice in qualitative analysis.

PART III

CHEMICAL ARITHMETIC

TYPES OF PROBLEMS

The first thing that is necessary in the working of any chemical problem is that the student should have clearly in mind the full significance of a chemical formula. From the text it is to be seen that the formula for potassium chlorate, KClO_3 , stands for one gram molecule of KClO_3 which is made up of one gram atom of potassium, one gram atom of chlorine and three gram atoms of oxygen. Before a quantitative problem can be worked these amounts must be converted to actual weight units, that is, we must think of KClO_3 as standing for a compound that is made up of 39.1 grams of potassium, 35.46 grams of chlorine and 3×16 grams or 48 grams of oxygen. When this conversion has been made, the material used in the experiment can be followed quantitatively from change to change through any series of *balanced* equations. All chemical problems fall under one of the following classes:

A. Problems Involving Weight Relationships Alone:

- I. Percentage Composition.
- II. Chemical Formula from Percentage Composition.
- III. Amount of One Substance Required to Combine with or Displace a given Amount of Another Substance.

B. Problems Involving Weight and Volume:

- IV. Specific Gravity of Solids and Liquids.
- V. The Correction of Gas Volumes for Temperature, Pressure and Water Vapor.
- VI. Gram-Molecular Volume.
- VII. Normal Solutions.

Before starting to work a problem analyze it carefully and classify it under one or more of the above types. It may be that the problem may involve the use of two or more of the above types in its solution.

I. Percentage Composition from Formula.

A formula represents a **molecule** or a **gram molecule** of a substance. It indicates the kind and number of atoms of which the molecule is composed. If the constituent atoms are all alike, the substance is an element — *e.g.*, O_2 , Cl_2 . If the atoms present in a molecule are those of different elements, the substance is a compound — *e.g.*, H_2O , $KClO_3$. Since to each element a definite relative (atomic) weight may be assigned, the percentage by weight of any element in a compound may be calculated from the formula of the compound.

MODEL EXAMPLE I

To calculate the per cent of oxygen in potassium chlorate.

Method of Solution. — The formula of potassium chlorate, $KClO_3$, shows that it contains an atomic weight of potassium (39), one of chlorine (33.5), and three atomic weights of oxygen (3×16). The sum of these numbers is 122.5 and is called the molecular weight of potassium chlorate. To express this number in actual weight units one may hence say that a gram molecule of potassium weighs (or is) 122.5 g. Of this weight 48 parts are oxygen. The percentage of oxygen in the compound is, therefore, the ratio to 100 which is equal to the ratio of 48 to 122.5.

That is, $x : 100 = 48 : 122.5$ and hence $x = 39.18$ per cent.

1. Compare the percentages of iron in ferrous oxide, ferric oxide, and magnetic oxide of iron. Which of the three is the most valuable iron ore? The formulas are: FeO , Fe_2O_3 , Fe_3O_4 .

2. (a) How many different kinds of atoms are there in each of the following substances: C_2H_5OH , $(NH_4)_2SO_4$, $C_{12}H_{22}O_{11}$, $Fe_2(SO_4)_3$?

(b) How many atoms are there in each of these molecules?

(c) Calculate the molecular weight of each substance.

3. Calculate the per cent of oxygen in each substance in 2 (a).

4. Calculate the percentage of water in crystallized "washing soda," $Na_2CO_3 \cdot 10H_2O$.

5. How many grams of sodium are present in 150 grams of sodium bicarbonate, $NaHCO_3$?

6. What weight of phosphorus is present in a kilogram of calcium phosphate, $Ca_3(PO_4)_2$?

II. Derivation of Formula from Percentage or Weight Composition.

When the composition of a substance is given in percentages or in actual weights, the ratio of the atoms can be determined and the **simplest** formula can thus be derived. If the molecular weight of the compound is known, the **actual** formula can be found for the compound.

MODEL EXAMPLE II

A compound contains 52.18 per cent carbon; 13.04 per cent hydrogen; and 34.78 per cent oxygen. What is its simplest formula?

Method of Solution. — The ratio of each kind of atom present is obtained by dividing the amounts of the elements by their respective atomic weights — thus:

$$\text{C} = \frac{52.18}{12} = 4.348; \text{divided by } 2.174 = 2$$

$$\text{H} = \frac{13.04}{1} = 13.04; \text{divided by } 2.174 = 6$$

$$\text{O} = \frac{34.78}{16} = 2.174; \text{divided by } 2.174 = 1$$

The first quotients which represent the atomic ratios must now be converted to small whole numbers. This is accomplished by dividing them by their highest common factor. It is apparent that the quotients obtained above are all multiples of 2.174 and hence the ratio of the atoms in the compound is $\text{C} : \text{H} : \text{O} = 2 : 6 : 1$ and the simplest formula is $\text{C}_2\text{H}_6\text{O}$. If this is the correct formula the molecular weight must be $2 \times 12 + 6 \times 1 + 16 = 46$. If, however, the molecular weight is some multiple of 46 (*e.g.*, 92) the formula must be multiplied accordingly (*e.g.*, $\text{C}_4\text{H}_{12}\text{O}_2$ for a mol. wt. of 92).

7. A compound having a molecular weight of 30 contains 80 per cent carbon and 20 per cent hydrogen. What is its formula?

8. Ten grams of iron when burned in oxygen form 13.81 g. of an oxide. Calculate the formula of the oxide. Assuming this to be the correct formula, calculate the molecular weight of the compound.

9. A compound by analysis gave 26.9 per cent sodium,

16.58 per cent nitrogen, and 56.52 per cent oxygen. Calculate its simplest formula.

10. A compound having a molecular weight of 106.5 is found to contain 21.6 per cent sodium, 33.3 per cent chlorine, and the remainder is oxygen. What is its simplest formula? Is this also its actual formula?

11. Derive the empirical formulas for the substances which have the following percentage compositions:

$$\begin{array}{ll} (a) \text{ K} = 26.59\% & (b) \text{ K} = 40.26\% \\ \text{Cr} = 35.39 & \text{Cr} = 26.77 \\ \text{O} = 38.02 & \text{O} = 32.95 \end{array}$$

12. Derive the empirical formula of the substance which gave the analysis: Ca = 18.28 per cent, Cl = 32.36 per cent, H₂O = 49.36 per cent.

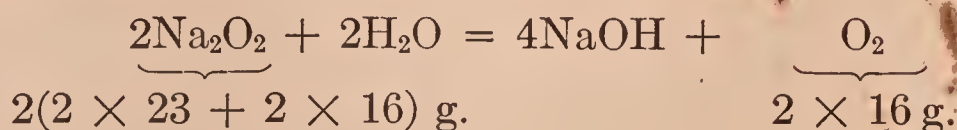
III. Simple Weight Calculation Based upon a Chemical Reaction.

In this type of problem the correct chemical equation must always be written first, and the calculation then based upon the weights of the substances involved in the reaction.

MODEL EXAMPLE III

What weight of oxygen may be obtained by the action of water upon 25 g. of sodium peroxide?

Method of Solution. — The equation,



shows that two gram molecules, 156 g., of sodium peroxide are required to produce one gram molecule, 32 g., of oxygen. Unless the equation had been written it might have been wrongly assumed that sodium peroxide would liberate all of its oxygen instead of only half of it. Since the equation shows that 156 g. of Na₂O₂ will give 32 g. of O₂, it must follow that 25 g. of Na₂O₂ will give a proportionate amount — *i.e.*,

$$\frac{156 \text{ g.}}{25 \text{ g.}} = \frac{32 \text{ g.}}{x \text{ g.}}; \text{ and } x = \frac{32 \times 25}{156} = 5.12 \text{ g. O}_2$$

Care must be taken to have the proportion correctly expressed. It should also be noted that only **two kinds** of quantities can

ever appear in any single proportion. In one ratio of the above proportion we have **grams of sodium peroxide**; in the other ratio we have **grams of oxygen**. The weight of water used in the reaction or the weight of sodium hydroxide produced could be calculated by constructing separate proportions for each.

13. What weight of mercuric oxide must be decomposed in order to obtain 50 g. of oxygen?

14. What weight of oxygen may be obtained from 100 g. of potassium chlorate?

15. What weight of potassium chloride is formed when 20 g. of oxygen are liberated from potassium chlorate?

16. What weight of oxygen is needed to burn completely 40 g. of phosphorus?

17. How many grams of oxygen are required to burn 160 g. of sulfur? How many gram atoms of oxygen is this? What weight of sulfur dioxide is formed? This weight of sulfur dioxide will combine with what weight of water?

18. What weight of magnetic oxide of iron is formed when 56 g. of iron are burned in oxygen? What weight of oxygen combines with 56 g. of iron?

19. How much zinc sulfate is formed when a gram of hydrogen is liberated by the action of sulfuric acid on zinc?

20. Calculate the weight of hydrogen obtained when 10 g. of water react with sodium. What weight of hydrogen is obtained from the same weight of water reacting (as steam) with iron?

21. Hydrogen is passed over hot magnetic oxide of iron until the latter has lost 3.2 g. What weight of metallic iron has been produced?

22. What weight of water is formed by reducing 80 g. of copper oxide with hydrogen? What weight of copper is left?

IV. Specific Gravity of Solids and Liquids.

The density (mass per unit volume) of solids and liquids is usually compared with that of water as the standard. The relative density compared with water is called the *specific gravity*. It is customary to make this comparison at the temperature at which water has its maximum density (4°C.) and at standard atmospheric pressure. When a body is heavier than

water, the specific gravity may be obtained by dividing its weight in air by its apparent loss of weight when weighed in water. If the substance is soluble in water, it may be weighed in a liquid in which it is insoluble and of which the specific gravity is known.

MODEL EXAMPLE IV

A piece of granite weighs 2.30 g. in air and 1.52 g. in water. Calculate the specific gravity of the granite.

Method of Solution. — The sample of granite displaces a volume of water equal to the volume of the sample of granite. The apparent loss in weight of the sample of granite is equal to the weight of the water displaced. Since 1 cc. of water weighs 1 g., the loss in weight of the granite is numerically equal to the volume of the water displaced which is, in turn, equal to the volume of the sample of granite. In the problem above, the loss in weight is 0.78 g., hence the volume of the granite is 0.78 cc. and its specific gravity is $2.30/0.78 = 2.948$.

23. What volume is occupied by 100 g. of HNO_3 if its specific gravity is 1.42?

24. A solution of hydrochloric acid has a specific gravity of 1.2 and contains 40 per cent by weight of HCl . Calculate the volume of this acid solution which will contain 50 g. of HCl .

25. A piece of glass weighs 25.86 g. in air, 15.26 g. in water, and 9.20 g. in sulfuric acid. Calculate the specific gravity of the glass and of the sulfuric acid.

26. A bottle weighs 50.50 g. When filled with water the bottle weighs 258.58 g., and when filled with oil it weighs 220.20 g. Calculate the specific gravity of the oil.

27. A graduate cylinder is filled with water to the 75-cc. mark. When 25 g. of rhombic sulfur are dropped into this, the water level rises to the 87.2-cc. mark. Calculate the specific gravity of rhombic sulfur.

28. What volume of sulfuric acid of specific gravity 1.84 and containing 94 per cent by weight of H_2SO_4 will be required to neutralize completely 50 cc. of sodium hydroxide solution which has a specific gravity of 1.35 and contains 31.5 per cent by weight of NaOH ?

V. Application of the Laws of Boyle and Charles — Calculation of Volume Change Caused by Change in Temperature and Pressure.

The volume occupied by a definite quantity of gas at a certain temperature and pressure may be calculated if its volume at any other temperature and pressure is given. It is only necessary to remember that the volume of a gas varies **directly** with the **absolute** temperature and **inversely** with the pressure to which it is subjected.

The volume occupied by a gas has no significance unless the temperature and pressure at which it is measured are also stated. For convenience a definite temperature and pressure have been agreed upon at which to compare gases. These are 0° Centigrade and 760 mm. pressure and are called **normal** or **standard conditions**. Unless others are specified, these conditions are understood when the volume of a gas is mentioned.

Gases are ordinarily measured over dry mercury because that liquid has a very low vapor pressure at ordinary temperatures. If a gas is measured over water, part of its apparent volume and pressure is due to water vapor, for which correction must be made in calculating the true volume of the gas.

MODEL EXAMPLE V

If a quantity of oxygen measures 250 cc. at 18° C. and 750 mm. pressure, what volume will it occupy at 25° C. and 780 mm. pressure?

Method of Solution. — First convert the temperatures to the Absolute scale by algebraically adding 273°. We can now apply the two gas laws. In the change indicated in the problem the temperature is increased and will increase the volume in the ratio of $\frac{273 + 25}{273 + 18}$. (For an increase, the larger number is placed on top.) The pressure is also increased but this will tend to decrease the volume in inverse ratio — *i.e.*, $\frac{750}{780}$. The two effects can be combined in the form:

$$250 \text{ cc.} \times \frac{298}{291} \times \frac{750}{780} = \quad ? \text{ cc. at the new conditions, } 25^{\circ} \text{ C., } 780 \text{ mm.}$$

If the gas were measured over water at 18°C . and 750 mm. and its **dry** volume at 25°C . and 780 mm. is desired, it is necessary to subtract the vapor pressure of water at 18° (refer to table) from the observed pressure of 750 mm. and to use the remainder in place of 750 in the above calculation.

29. If a volume of gas measures 1 l. at 0°C ., what will be its volume at 100°C . if the pressure remains constant?

30. If the volume of a gas measures 400 cc. at 750 mm. pressure, calculate its volume if the pressure is changed to 500 mm., assuming constant temperature.

31. A volume of air measuring 200 cc. at 20°C . and 700 mm. pressure will become what volume at -20°C . and 800 mm. pressure?

32. Calculate the volume at standard conditions occupied by:

(a) 500 cc. of a gas measured at 22°C . and 640 mm. pressure.

(b) 310 cc. of a gas measured at -15°C . and 900 mm. pressure.

(c) 25 l. of gas measured at 100°C . and 100 mm. pressure.

33. Twenty liters of hydrogen at standard conditions will occupy what volume over water at 26°C . and 765 mm. pressure?

34. If an automobile tire has a volume of 1.2 cu. ft. when inflated, how many cu. ft. of air, measured at standard conditions, must be pumped into it in order to give a pressure of 3 atmospheres at 25°C .? An atmosphere of pressure is 760 mm. of mercury or 15 lb. per square inch.

35. What volume of hydrogen at standard conditions will be liberated from a gram molecule of sulfuric acid by zinc? What volume will this hydrogen occupy at 20°C . and 870 mm. pressure?

36. If 430 cc. of a gas at 20°C . and 780 mm. pressure weigh 1.54 g., what is the weight of a liter of the gas at standard conditions?

37. Oxygen was collected over water when the barometric pressure was 735 mm. and the temperature was 22°C . What would be the true volume of 200 cc. of this gas at standard conditions?

38. What weight of zinc would be required to displace enough hydrogen from an acid to inflate a balloon having a capacity of 500 liters at 20°C . and 800 mm. pressure?

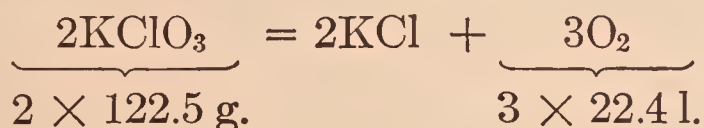
VI. Problems Involving the Gram-Molecular Volume of Gases.

It follows from Avogadro's Law that gram molecules of different gases will occupy equal volumes. The volume of a gram molecule of any gas at standard conditions is 22.4 liters. Hence the molecular formula of any gas has a volume significance as well as one of definite weight — see Type III. For example, the formula O_2 may be read 32 g. of oxygen or 22.4 l. of oxygen (standard conditions); the formula CO_2 represents 44 g. and also 22.4 l. of carbon dioxide gas.

MODEL EXAMPLE VI

What volume of oxygen at standard conditions may be obtained from 50 g. of potassium chlorate?

Method of Solution. — This problem, like Model III, is based upon a chemical reaction, so a balanced equation should first be written:



This shows that 245 g. of potassium chlorate will liberate 67.2 l. of oxygen. A proportionate volume of oxygen may be obtained from 50 g. of the chlorate — *i.e.*,

$$\frac{245}{50} = \frac{67.2}{x}; x = 13.8 \text{ l. of oxygen.}$$

(*Caution:* It must be noted that the gram-molecular volume (G.M.V.) idea, like Avogadro's Law, applies only to substances in the gaseous state. Also, care must be taken to employ the correct molecular formula for the gas. Thus, H_2 , and not H, represents 22.4 l. of hydrogen.)

39. If 90 g. of water are decomposed by electrolysis, what volume of hydrogen is obtained? What volume and weight of oxygen? If the same weight of water reacts with sodium, what volume of hydrogen is obtained?

40. What weight of iron will be produced in the reduction of its magnetic oxide by means of 24 l. of hydrogen?

41. What volume of ozone may be obtained from 75 l. of oxygen if the conversion is 8 per cent efficient? What will this volume of ozone weigh?

42. What is the numerical value of the G.M.V. at 25° C. and 750 mm. pressure?

43. How many grams of oxygen are needed to burn completely 60 g. of carbon? How many gram atoms of oxygen is this? How many gram molecules? What volume does the product occupy at standard conditions?

44. What is the weight of a liter of a gas having the formula C_2H_6 ? What is its density compared with air?

45. If 400 cc. of a gas weigh 0.520 g., what is the molecular weight of the gas?

46. If an electric spark is passed through a mixture of 524 cc. of hydrogen and 275 cc. of oxygen, will any gas remain after the tube is cooled? If so, what gas and how much?

47. What volume of hydrogen must be burned in chlorine in order to make enough hydrochloric acid to neutralize 60 g. of sodium hydroxide?

48. Compare the volumes of chlorine produced when hydrochloric acid reacts with 50 g. of manganese dioxide and when it reacts with 50 g. of potassium permanganate.

x 49. What volume of chlorine may theoretically be liberated from a kilogram of bleaching powder by the action of sulfuric acid?

50. What volume of chlorine, reacting with a dilute solution of KOH, is needed to prepare 12 g. of potassium hypochlorite? What volume of chlorine, reacting with a concentrated solution of KOH, is needed to prepare 12 g. of potassium chlorate? In each case what per cent of the chlorine used appears in the product named?

51. What volume of bromine vapor, measured at $25^\circ C.$ and 780 mm. pressure, may be formed from 200 cc. of liquid bromine? The density of liquid bromine is 3.18.

52. What volume of hydrogen bromide is obtained when 10 g. of phosphorus are combined with bromine and the product hydrolyzed with water?

53. A 10 per cent solution of potassium iodide has a density 1.08. What volume of chlorine is needed to displace the iodine in a liter of this solution?

x 54. What volume of hydrogen fluoride, H_2F_2 , is needed to react with 20 g. of $CaSiO_3$? What weight of calcium fluoride is required to prepare this quantity of hydrogen fluoride?

55. Calculate the weight of ferrous sulfide needed to prepare enough H_2S to fill a gasometer having a capacity of 2 cu. m. at $25^\circ C.$ and 750 mm. pressure.

56. What weight of antimony sulfide is formed when 2 l. of H_2S are passed into a solution of antimony chloride?

57. If a solution of $\text{K}_2\text{Cr}_2\text{O}_7$ is acidified with HCl and 5 l. of H_2S gas are used, what weight of $\text{K}_2\text{Cr}_2\text{O}_7$ will be reduced?

58. What weight of copper is needed to prepare 10 l. of sulfur dioxide by the reduction of concentrated sulfuric acid? What weight of sodium sulfite may be prepared with this quantity of the gas?

59. What volume of sulfur dioxide will, when dissolved in water, reduce 30 g. of potassium permanganate?

60. Compare the volumes of sulfur dioxide obtainable from 5 g. of Na_2SO_3 and from 5 g. of $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$ by the action of an acid.

61. What volume of hydrogen will combine with 30 l. of nitrogen in the Haber synthesis of ammonia? What volume of ammonia will be produced by this combination?

62. What volume of ammonia is liberated by the action of lime upon a kilogram of ammonium sulfate? By the hydrolysis of a kilogram of calcium cyanamide?

VII. Equivalent Weights and Normal Solutions.

The following problems deal with solutions of definite strength, expressed in terms of **normality** — that is, the number of gram equivalents of the solute in a liter of the solution. The normality idea may involve:

(a) The weight of a substance and the volume and strength of a solution.

(b) The volumes and strengths of two solutions.

(c) The volume of a gas and the volume and strength of a solution.

MODEL EXAMPLE VII

(a) What weight of pure H_2SO_4 is contained in 400 cc. of 1.2 N sulfuric acid solution?

(b) If 35 cc. of this solution just neutralize 35 cc. of a basic solution, what is the normality of the latter?

(c) What volume of CO_2 gas will be liberated by the action of 200 cc. of this 1.2 N acid on marble?

Method of Solution. — (a) A gram molecule of sulfuric acid (98 g.) contains 2 gram equivalents. In a liter of normal sulfuric acid there will hence be 49 g. of H_2SO_4 . In a liter of 1.2 N

Normal solution = 1 gm eq

sulfuric acid there will be $49 \times 1.2 = 58.8$ g. In 400 cc. of 1.2 N sulfuric acid there will be $58.8 \times \frac{400}{1000} = 23.52$ g. of H_2SO_4 .

(b) It is apparent that 1 cc. of the base is equivalent to 35/25, or 1.4 cc. of the acid. That is, the base is 1.4 times as strong as the acid. The acid is 1.2 N, so that the base must be $1.2 \text{ N} \times 1.4 = 1.68 \text{ N}$.

It may be noted that the product of normality $\times$ volume of any solution = the product of normality $\times$ volume of an equivalent amount of any other solution. That is, $NV = N'V'$. If three factors in this equation are known, the fourth may be found. In the above problem the unknown is the normality (N') of one solution. Using the equation to solve this problem,

$$1.2 \times 35 = N' \times 25; N' = \frac{1.2 \times 35}{25} = 1.68 \text{ N}.$$

(c) The chemical action involved may be expressed by the equation:



This shows that 2 l. of N (or 1 l. of 2 N) H_2SO_4 will liberate a gram-molecule, 22.4 l. of CO_2 gas. It follows that 1 l. of N acid will liberate 11.2 l. of CO_2 ; that 1 l. of 1.2 acid will liberate $11.2 \times 1.2 = 13.44$ l. of CO_2 ; and hence 200 cc. of 1.2 acid will liberate $13.44 \times \frac{200}{1000} = 2.688$ l. or 2,688 cc. of CO_2 .

63. A gram molecule of $\text{Al}_2(\text{SO}_4)_3$ will make how many liters of N/5 solution? What weight of the salt will be present in a liter of this solution?

64. The specific gravity of a 20 per cent solution of hydrochloric acid is 1.1. What is the normality of the solution?

65. How many grams of the following substances will be present in a liter of normal solution of each: H_3AsO_4 , SnCl_4 , $\text{Fe}(\text{NO}_3)_3$?

66. How many gram equivalents of Na_2SO_4 are needed to prepare 5 l. of 2 N sodium sulfate solution? How many grams of sodium sulfate will this be?

67. What volume of 1.5 N base will be needed to neutralize 3 l. of 6 N acid?

68. What weight of sodium hydroxide is needed to neutralize 4 l. of 1.5 N sulfurous acid? What volume of 0.2 N solution will this weight of NaOH prepare?

1.2
0.4

69. If 500 cc. of an acid liberate 1.1 l. of SO_2 from a sulfite, what is the normality of the acid?

70. If a gram molecule of SO_2 is dissolved in enough water to make 10 l. of sulfurous acid solution, what is the normality of the solution?

71. What weight of lead sulfide may be precipitated from a solution of lead nitrate by the action of 500 cc. of $\text{N}/5 \text{ H}_2\text{S}$ solution?

72. What volume of ammonia gas will be liberated from an ammonium salt by the action of 3 l. of 1.3 N alkali?

73. What volume of ammonia gas is required to neutralize 300 cc. of 2.5 N phosphoric acid?

74. If the ammonia resulting from the action of a base on 4 g. of ammonium nitrate is dissolved to make 500 cc. of ammonium hydroxide, what is the normality of the solution?

MISCELLANEOUS EXERCISES

VALENCE EXERCISES

75. Indicate the valence of the metal and of the acid radical in each of the following: NaNO_3 , MgCO_3 , AlCl_3 , $\text{Ca}_3(\text{PO}_4)_2$, K_2SO_4 , Bi_2S_3 , AgClO_3 , $\text{Cr}_2(\text{SO}_4)_3$. Name each compound.

76. Using the valences found in the preceding problem, write the formulas for: calcium nitrate, sodium sulfide, aluminum phosphate, chromium chlorate, bismuth chloride, silver sulfate, potassium carbonate, magnesium chlorate.

77. Write formulas for the salts formed by the action of nitric, sulfurous and phosphoric acids on each of the following bases: KOH , FeO , $\text{Fe}(\text{OH})_3$, $\text{Ca}(\text{OH})_2$. Name the twelve salts.

78. If mercury and tin form the oxides: HgO , Hg_2O and SnO , SnO_2 , write the formulas for mercurous nitrate, mercuric chlorate, stannic chloride and stannous sulfate.

* * *

79. What weight of copper must react with nitric acid to produce 30 l. of nitric oxide?

80. What volume of nitrogen dioxide will be formed by the action of concentrated nitric acid on 12 g. of copper?

81. What weight of ammonium nitrate can be obtained by neutralizing 3 l. of 1.4 N nitric acid with ammonia? What volume of nitrous oxide can be obtained from this weight of ammonium nitrate?

82. What weight of nitric acid is present in a liter of its solution, specific gravity 1.42, containing 70 per cent HNO_3 ? What volume of ammonia will this weight of nitric acid neutralize?

83. What weight of carbon is required to reduce enough bone ash, mixed with sand, to form 12 g. of phosphorus?

84. What volume of phosphine can be prepared by the action of 400 cc. of 3 N KOH on phosphorus?

85. What volume of chlorine gas will combine with 6 g. of phosphorus to form the pentachloride? What volume of N HCl solution may be prepared by the hydrolysis of this product?

86. What volume of N/2 H_2S solution will be required to precipitate 8 g. of antimony sulfide from a solution of SbCl_3 ?

87. What weight of Na_2CO_3 and what volume of 3 N H_2SO_4 must be mixed to produce 2 l. of carbon dioxide?

88. Calculate the volume of N/4 calcium hydroxide that will react with 2 l. of carbon dioxide.

89. What volume will 20 g. of CO_2 occupy at 25°C . and 775 mm. pressure?

90. What weight of magnesium is needed to reduce 6 g. of silica to magnesium silicide?

91. Calculate the weight of a G.M.V. of air assuming it to be a mixture of: nitrogen, 78 per cent; oxygen, 21 per cent; argon (molecular weight 40), 1 per cent.

92. If a gas has a density of 1.24 referred to air, calculate the weight of a liter of the gas and its molecular weight.

93. If 654 cc. of hydrogen, measured at 23°C . and 745 mm. pressure, are liberated from 200 cc. of sulfuric acid solution by the action of magnesium, calculate the normality of the sulfuric acid.

94. Calculate the percentage of water of hydration in borax. What weight of crystalline borax must be treated with an acid to prepare 5 g. of boric acid?

95. If a certain ore contains 30 per cent cuprous oxide, what weight of the ore must be worked in order to obtain a ton of pure copper?

96. What volume of sulfur dioxide is obtained when a kilogram of iron pyrites is roasted? What volume of oxygen is required? What volume of air? What weight of carbon will be required to reduce the iron oxide which is produced from a kilogram of iron pyrites?

97. The specific heat of platinum is 0.0324, and a gram atom of chlorine combines with 48.6 g. of platinum to form platinic chloride. Calculate (a) the atomic weight of platinum and (b) the formula of platinic chloride.

98. What volume of hydrogen is obtained when 10 g. of magnalium alloy (30 Mg, 70 Al) dissolve in sulfuric acid?

99. What weight of pure nitric acid is required to dissolve 100 g. of coinage alloy (90 Ag, 10 Cu) assuming that nitric oxide is formed in the reactions? What volume of 5 N nitric acid solution would this be?

100. What weight of microcosmic salt must be heated to prepare 5 g. of sodium metaphosphate? What volume of ammonia is liberated at the same time?

101. What volume of arsine will be formed if a gram of As_2O_3 is dissolved in HCl and added to a hydrogen generator?

102. What volume of carbon dioxide can be obtained (a) by heating 42 g. of sodium bicarbonate? (b) by treating the same weight of the salt with an acid?

103. Calculate the weight of caustic soda and the volumes of chlorine and hydrogen which may be obtained by the electrolysis of a solution containing a kilogram of common salt.

104. Twenty cc. of a sodium carbonate solution required 50 cc. of N/10 hydrochloric acid for neutralization to the phenolphthalein end-point. What volume of this acid would have been required if methyl orange had been used as the indicator? What weight of Na_2CO_3 is contained in a liter of this solution?

105. A mixture of Na_2CO_3 and NaHCO_3 was dissolved in water and titrated with normal hydrochloric acid. To reach the phenolphthalein end-point 20 cc. of the acid were required. Methyl orange was then added and an additional 30 cc. of normal hydrochloric acid was used to cause this indicator to change color. Calculate the weights of Na_2CO_3 and NaHCO_3 in the mixture.

106. Write the equations for the production of potassium chlorate from potassium chloride by the electrolytic method. Calculate the weight of the chlorate which can theoretically be obtained from a kilogram of sylvite by this method. What volume of hydrogen would be obtained as a by-product?

107. Calculate the normality of concentrated ammonium hydroxide which has a specific gravity of 0.90 and is 28 per

cent ammonia. Calculate the volume of ammonia gas which would be required to prepare a liter of this solution.

108. Starting with marble give equations and brief statements to show how the following could be obtained: (a) quicklime, (b) limewater, (c) calcium bicarbonate, (d) calcium chloride, (e) metallic calcium, (f) gypsum, (g) plaster of Paris.

109. One gram of calcite gave 210 cc. of carbon dioxide (standard conditions) when treated with an acid. What was the percentage of calcium carbonate in the sample of calcite?

110. The addition of barium chloride solution to 250 cc. of dilute sulfuric acid produced a precipitate which weighed 3.90 g. Calculate the normality of the acid.

111. From solubility relations explain how the following results are possible: (a) The filtrate from BaCO_3 gives a precipitate with potassium dichromate solution and with sulfuric acid, the filtrate from CaCO_3 does neither. (b) The filtrate from CaSO_4 gives a precipitate with ammonium oxalate solution, but the filtrate from SrSO_4 does not.

112. Compare the alkali elements and their compounds with those of the alkaline earth family. This may be done in tabular form. Suggest a precipitation method for separating calcium, strontium, and barium from a mixture which contains also salts of sodium, potassium and ammonium.

113. If given 100 g. of silver coin (90 Ag, 10 Cu) and told to return the silver as silver chloride, how would you proceed and what weight of silver chloride should you return?

114. An electric current flows through three cells in series: Cell A contains a solution of cupric sulfate; B, a solution of silver nitrate; and C, dilute sulfuric acid. What weights of copper and silver are deposited respectively in A and B while 3 l. of a mixture of hydrogen and oxygen are being evolved from C?

115. In a Daniell cell what weight of copper has been deposited when 5 g. of zinc have dissolved?

116. Write the equation by which magnesia could be dissolved, precipitated and ignited to form magnesium pyrophosphate. If a gram sample of insulating material when treated in this manner gave 2.35 g. of magnesium pyrophosphate, calculate the percentage of magnesia in the material.

117. Calculate the volume of carbon dioxide which may be obtained from a kilogram of dolomite. Show by equations how

you could start with dolomite, dissolve it, remove the calcium as the carbonate, and obtain metallic magnesium from the filtrate.

118. Write the equations by which zinc may be obtained from zinc blende. What weight of zinc and what weight of sulfuric acid (by-product) can be obtained from a ton of ore which is 30 per cent ZnS ?

119. Calculate the weight of aluminum which should be added to 16 g. of ferric oxide to make a thermite mixture. Calculate the number of calories liberated when this amount of the mixture reacts.

120. Outline the tests by which you would distinguish the following from each other: Aluminum sulfate, zinc sulfate, cadmium sulfate, mercuric sulfate, mercurous sulfate.

121. Construct a series of equations by which bauxite may be converted to alum. What weight of alum may be produced from a metric ton of bauxite if the ore contains 50 per cent Al_2O_3 ?

122. By means of ionic equilibria show what happens when a solution of sodium carbonate is added to a solution of aluminum sulfate.

123. Starting with tin in each case, write equations to show how the following may be prepared: (a) Sodium stannite; (b) sodium stannate; (c) ammonium sulfostannate.

124. The sulfides PbS , CuS , HgS , SnS when precipitated are dark brown to black in color. Briefly outline chemical tests by which each could be distinguished from the others.

125. Calculate the volume of chlorine, measured at 20°C. , and 740 mm. pressure, which is required to react completely with 2 g. of tin.

126. Outline the process by which lead is obtained from galena. What volume of concentrated sulfuric acid (sp. gr. = 1.84; 95 per cent H_2SO_4) could be manufactured from the sulfur dioxide evolved when a kilogram of galena is roasted?

127. What chemical test would you make to decide whether PbO_2 is a dioxide or a peroxide? Explain.

128. Construct the balanced equation for the oxidation of ferrous sulfate by potassium dichromate in presence of excess sulfuric acid. Calculate the weight of potassium dichromate which must be present in a liter of solution so that 1 cc. of the solution is equivalent to (will oxidize) 10 mg. of ferrous iron.

129. Starting with chromic hydroxide show by equation how

you could obtain: (a) a green solution; (b) a yellow solution; (c) a yellow precipitate; (d) an orange solution.

130. In order to separate chromium and aluminum the following procedure may be used: (a) To a solution containing the two metals as chlorides add NaOH; (b) add NaOH and Na_2O_2 ; (c) acidify; (d) add NH_4OH . Write a pair of equations for each step and indicate how the two metals may be separated.

131. Calculate the volume of sulfur dioxide required to reduce 10 g. of potassium dichromate in the presence of excess sulfuric acid. Compare the volume of hydrogen sulfide required for the same purpose.

132. A quantity of solid potassium dichromate was heated with concentrated hydrochloric acid and the gas which was produced was passed into a solution of potassium iodide; if 2.4 g. of iodine were liberated, what weight of potassium dichromate must have been used?

133. A gram of pyrolusite ore was treated with hydrochloric acid and the evolved gas liberated 2.1 g. of iodine from an iodide. Calculate the per cent of manganese dioxide in the ore.

134. Show by equations how the following substances may be prepared from manganese dioxide: (a) manganese; (b) ferromanganese; (c) potassium manganate; (d) potassium permanganate; (e) manganous sulfate.

135. What volume of potassium permanganate solution, containing a gram mole of the salt per liter, is required to oxidize 50 cc. of N/4 sulfurous acid?

136. An oxide of iron contains 27.6 per cent oxygen; what is its formula? If an ore contains 90 per cent of this oxide and 10 per cent silica, write equations to show how pure iron may be obtained from the ore.

137. How would you prepare a solution of potassium permanganate so that 1 cc. of the solution is equivalent to 10 mg. of ferrous iron?

138. How would you prepare: (a) ferric chloride from iron; (b) ferrous sulfide from ferric chloride; (c) ferric sulfate from ferrous sulfide? Write the equations.

139. Given a mixture of ferric chloride and aluminum chloride, how could the metals be separated by means of a single reagent? Write the equations. Repeat the problem for a mixture of ferrous sulfate and copper sulfate.

140. What weight of pure iron will be dissolved by 2 l. of 6 N sulfuric acid? Calculate the volume occupied by the liberated hydrogen at 25° C. and 780 mm. pressure.

141. If 50 cc. of ferrous sulfate solution, containing excess sulfuric acid, reduced 1.58 g. potassium permanganate, what was the normality of the solution with respect to ferrous sulfate?

142. Give the equations and the practical details for the preparation of pure crystals of ferrous ammonium sulfate from iron pyrites.

143. Starting with ferrous chromite give the equations and details by which you would get the iron and chromium into separate solutions.

144. Pyrolusite ore contains ferric oxide and silica as impurities. Outline a procedure by which you could obtain a pure manganese compound from the ore.

145. If 10 g. of pure ferric oxide are converted to a solution of the sulfate, what volume of concentrated ammonium hydroxide (sp. gr. = 0.90; 28 per cent NH_3) must be added to form ferric ammonium alum?

146. A solution gave a white precipitate with HCl ; the precipitate turned black when NH_4OH was added. The filtrate gave a black precipitate with H_2S which was insoluble in HNO_3 but soluble in aqua regia.

The solution in aqua regia, treated with SnCl_2 , gave a white precipitate which changed to gray. The filtrate from the black precipitate gave a flocculent white precipitate when treated with $(\text{NH}_4)_2\text{S}$. This precipitate was soluble in NaOH but not appreciably soluble in NH_4OH . What ions were present in the original solution? Write equations for the reactions which occurred.

147. Calculate the weight of impure sodium nitrate, containing 85 per cent NaNO_3 , required to produce 5 liters of nitric acid of specific gravity 1.45 and containing 77 per cent HNO_3 by weight.

148. A solution of nitric acid of specific gravity 1.25 contains 39.82 per cent HNO_3 by weight. Calculate the normality of the solution and the weight of HNO_3 in 10 cc. of the solution.

149. The analysis of a sample of ferric oxide showed that in 2.5 g. of the oxide there were 1.72 g. of iron. Calculate the percentage purity of the ferric oxide.

150. A solution of potassium hydroxide containing 40 per cent by weight of KOH has a specific gravity of 1.4. Calculate the weight of potassium chlorate which may be obtained by passing an excess of chlorine gas into 600 cc. of this solution.

APPENDIX

THE METRIC SYSTEM

Units		
METER	LITER	GRAM
<i>Prefixes</i>		
		<i>Meaning</i>
Kilo.....		1000
Hecto.....		100
Deka.....		10
Deci.....		.1
Centi.....		.01
Milli.....		.001

RELATION OF METRIC AND ENGLISH
UNITS

1 meter	= 39.3709 inches
1 kilogram	= 2.2046 pounds (Av.)
1 gram	= 15 grains
1 liter	= 1.0567 liquid quarts (U. S.)
1 liter	= 0.9081 dry quart
1 liter	= 0.8798 Imperial quart (Brit.)
1 mile	= 1.61 kilometers
1 pound (Av.)	= 454 grams
1 pound (Tr.)	= 373 grams

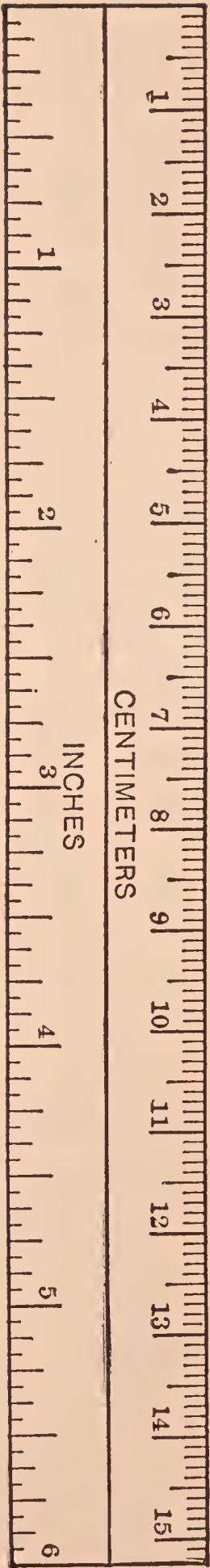


FIG. 29

ELECTROMOTIVE SERIES

THESE METALS LIBERATE HYDROGEN FROM:

These metals NOT precipitated as sulfides.	Potassium Sodium Barium Strontium Calcium	Cold Water	violently
These metals precipitated as sulfides in alkaline solution (Except Cr)	Magnesium Aluminum Manganese Zinc Chromium Cadmium Iron Cobalt Nickel	Steam — when heated	ACIDS — such as HCl and H ₂ SO ₄
	Tin Lead		slowly
These metals precipitated as sulfides in acid solution	Hydrogen Copper Arsenic Bismuth Antimony Mercury Silver Palladium Platinum Gold	Do not liberate Hydrogen	INACTIVE

APPROXIMATE ATOMIC WEIGHTS

	SYMBOL	ATOMIC WEIGHT		SYMBOL	ATOMIC WEIGHT
Aluminum . . .	Al	27.0	Iron	Fe	56.0
Antimony . . .	Sb	121.0	Lead	Pb	207.0
Arsenic	As	75.0	Magnesium . . .	Mg	24.0
Barium	Ba	137.0	Manganese . . .	Mn	55.0
Bismuth	Bi	208.0	Mercury	Hg	200.0
Boron	B	11.0	Nickel	Ni	58.5
Bromine	Br	80.0	Nitrogen	N	14.0
Calcium	Ca	40.0	Oxygen	O	16.0
Carbon	C	12.0	Phosphorus . . .	P	31.0
Chlorine	Cl	35.5	Platinum	Pt	195.0
Chromium . . .	Cr	52.0	Potassium	K	39.0
Cobalt	Co	59.0	Radium	Ra	226.0
Copper	Cu	63.5	Silicon	Si	28.0
Fluorine	F	19.0	Silver	Ag	108.0
Gold	Au	197.0	Sodium	Na	23.0
Helium	He	4.0	Sulfur	S	32.0
Hydrogen	H	1.0	Tin	Sn	119.0
Iodine	I	127.0	Zinc	Zn	65.0

VAPOR PRESSURE OF WATER

TEMPERATURES	PRESSURE IN MM. OF MERCURY	TEMPERATURE	PRESSURE IN MM.
10°	9.2	25°	23.5
11°	9.8	26°	25.0
12°	10.5	27°	26.5
13°	11.2	28°	28.1
14°	11.9	29°	29.8
15°	12.7	30°	31.6
16°	13.6	31°	33.4
17°	14.5	32°	35.4
18°	15.4	33°	37.4
19°	16.4	34°	39.6
20°	17.4	35°	41.9
21°	18.5		
22°	19.7	99°	733.2
23°	20.9	100°	760.0
24°	22.2	101°	787.6

FIRST AID INSTRUCTIONS

Minor Cuts and Bruises. — Flush wound with water, dry with sterile gauze, apply mercurochrome solution (use sparingly), bandage with roller gauze.

Severe Bleeding. — If from a cut artery, stop with pledget of gauze directly on leak or apply a tourniquet (towel or rubber tube) between wound and heart. Coagulation of blood may be hastened by application of pledget of gauze moistened with aluminum acetate. Bandage with gauze and roller bandage. CAUTION, NEVER APPLY COTTON TO AN OPEN WOUND.

Burns, Steam and Hot Water. Apply cold running water at once. When affected parts have been cooled remove clothing at your leisure, dry, apply butesin picrate and bandage loosely if necessary.

Burns, Fire, Hot Objects, etc. — Apply butesin picrate and bandage if necessary.

Burns by Chemicals.

Acids. — Flood with water, apply saturated solution of sodium bicarbonate.

Alkalies. — Flood with water. Apply boric acid solution or 1 to 2 per cent acetic acid.

Bromine. — Flood with water. Apply sodium bicarbonate solution or sodium thiosulfate solution. Keep wound moist with glycerine.

Phosphorus. — Flood with water. Apply mixture of sodium bicarbonate and hydrogen peroxide.

Poisons Inhaled as Gases and Fumes — General Instructions.

— Secure fresh air and have patient breathe deeply. Do not stimulate circulation by rubbing and chafing.

Acid Vapors. — Counteract with inhalation of dilute ammonia.

Hydrogen Sulfide Gas. — Give fresh air. Inhale ammonia.

Chlorine and Bromine Gas. — Give fresh air. Inhale vapors of alcohol or, better still, inhale vapors of tincture of benzoin.

Fainting. — Secure fresh air, recline patient, inhale ammonia (smelling salts). In more severe cases heart action may be stimulated by aromatic spirits of ammonia.





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